WEST Search History

DATE: Saturday, May 03, 2003

Set Name side by side	Query	Hit Count	Set Name result set
DB=USPT,PGPB; PLUR=YES; OP=OR			
L14	L13 or l12 or l11	38	L14
L13	L4 and l6	34	L13
L12	L4 and l7	25	L12
L11	L10 and l4	19	L11
L10	L9 and l5	64	L10
L9	L8 and l6	133	L9
L8	L7 and (radical)	850	L8
L7	(spin near3 (trap$6 or label$5))	1722	L7
L6	nitron$6	2731	L6
L5	PBN	907	L5
L4	L3 or l2 or l1	2656	L4
L3	((564/282)!.CCLS.)	309	L3
L2	((562/62)!.CCLS.)	34	L2
L1	((315,399,422,428,517,518,645,708,709/)!.CCLS. |(514/400 |514/654 |514/644 |514/641 |514/619 |514/643 |514/226.2)!.CCLS.)	2344	L1

END OF SEARCH HISTORY

```
* * * * * * * * * * * * * * STN Columbus * * * * * * * * * * * * * * * *


FILE 'HOME' ENTERED AT 11:11:52 ON 03 MAY 2003

=> file reg
COST IN U.S. DOLLARS                              SINCE FILE      TOTAL
                                                       ENTRY    SESSION
FULL ESTIMATED COST                                     0.21       0.21

FILE 'REGISTRY' ENTERED AT 11:12:06 ON 03 MAY 2003
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STRUCTURE FILE UPDATES:     2 MAY 2003  HIGHEST RN 509953-09-7
DICTIONARY FILE UPDATES:    2 MAY 2003  HIGHEST RN 509953-09-7

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 6, 2003

  Please note that search-term pricing does apply when
  conducting SmartSELECT searches.

Crossover limits have been increased.  See HELP CROSSOVER for details.

Experimental and calculated property data are now available.  See HELP
PROPERTIES for more information.  See STNote 27, Searching Properties
in the CAS Registry File, for complete details:
http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf

=> e 66893-81-0/cn
E1             1      6688: PN: WO9514772 SEQID: 6683 CLAIMED SEQUENCE/CN
E2             1      6689 PN: WO0118542 TABLE: 3A-1 CLAIMED DNA/CN
E3             0 --> 66893-81-0/CN
E4             1      6689: PN: EP1281758 SEQID: 6720 UNCLAIMED DNA/CN
E5             1      6689: PN: WO0078341 PAGE: 79 UNCLAIMED SEQUENCE/CN
E6             1      6689: PN: WO0153836 TABLE: 3-3 CLAIMED DNA/CN
E7             1      6689: PN: WO0164835 SEQID: 13689 CLAIMED DNA/CN
E8             1      6689: PN: WO0164835 SEQID: 20590 CLAIMED PROTEIN/CN
E9             1      6689: PN: WO0164835 SEQID: 27689 CLAIMED PROTEIN/CN
E10            1      6689: PN: WO0164835 SEQID: 6389 CLAIMED SEQUENCE/CN
E11            1      6689: PN: WO0177384 SEQID: 381689 CLAIMED DNA/CN
E12            1      6689: PN: WO9514772 SEQID: 6684 CLAIMED SEQUENCE/CN

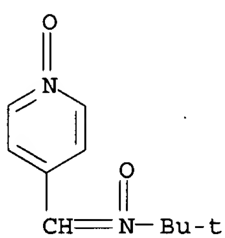
=> e POBN/cn
E1             1      POBILUKAST/CN
E2             1      POBILUKAST EDAMINE HYDRATE/CN
E3             1 --> POBN/CN
E4             1      POBO/CN
E5             1      POBR PROTEIN (BRUCELLA MELITENSIS BIOVAR SUIS STRAIN 1330 GE
                      NE POBR)/CN
E6             1      POBR REGULATOR (PSEUDOMONAS SP. STRAIN HR199(DSM-7063) GENE
                      POBR)/CN
E7             1      POBR REGULATOR (XANTHOMONAS AXONOPODIS CITRI STRAIN 306 GENE
                       POBR)/CN
E8             1      POBR REGULATOR (XANTHOMONAS CAMPESTRIS CAMPESTRIS STRAIN ATC
                      C33913 GENE POBR)/CN
E9             1      POC 202/CN
E10            1      POC 2020/CN
```

```
E11           1     POC MEDIUM/CN
E12           1     POC-AS 0010/CN

=> s e3
L1            1 POBN/CN

=> d l1

L1   ANSWER 1 OF 1  REGISTRY  COPYRIGHT 2003 ACS
RN   66893-81-0  REGISTRY
CN   2-Propanamine, 2-methyl-N-[(1-oxido-4-pyridinyl)methylene]-, N-oxide (9CI)
     (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN   2-Propanamine, 2-methyl-N-(4-pyridinylmethylene)-, N,N'-dioxide
OTHER NAMES:
CN   .alpha.-(4-Pyridyl-1-oxide)-N-tert-butylnitrone
CN   4-POBN
CN   C-(4-Pyridinyl-N-oxide)-N-tert-butylnitrone
CN   N-tert-Butyl-.alpha.-(4-pyridyl-1-oxide) nitrone
CN   POBN
FS   3D CONCORD
DR   83016-64-2
MF   C10 H14 N2 O2
CI   COM
LC   STN Files:   AGRICOLA, BEILSTEIN*, BIOSIS, CA, CAPLUS, CASREACT, CHEMCATS,
       CHEMLIST, CSCHEM, MSDS-OHS, NIOSHTIC, TOXCENTER, USPATFULL
         (*File contains numerically searchable property data)
     Other Sources:   EINECS**
         (**Enter CHEMLIST File for up-to-date regulatory information)
```



```
**PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT**

             144 REFERENCES IN FILE CA (1957 TO DATE)
              12 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
             143 REFERENCES IN FILE CAPLUS (1957 TO DATE)


=> e 66893-81-0/rn
E1            1     66893-79-6/RN
E2            1     66893-80-9/RN
E3            1 --> 66893-81-0/RN
E4            1     66893-82-1/RN
E5            1     66893-83-2/RN
E6            1     66893-84-3/RN
E7            1     66893-85-4/RN
E8            1     66893-86-5/RN
E9            1     66893-87-6/RN
E10           1     66893-88-7/RN
E11           1     66893-89-8/RN
E12           1     66893-90-1/RN
```

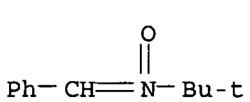
```
=> s e3
L2           1 66893-81-0/RN

=> e 3376-24-7/rn
E1           1      3376-14-5/RN
E2           1      3376-23-6/RN
E3           1 --> 3376-24-7/RN
E4           1      3376-25-8/RN
E5           1      3376-26-9/RN
E6           1      3376-27-0/RN
E7           1      3376-29-2/RN
E8           1      3376-30-5/RN
E9           1      3376-32-7/RN
E10          1      3376-33-8/RN
E11          1      3376-34-9/RN
E12          1      3376-35-0/RN

=> s e3
L3           1 3376-24-7/RN

=> d l3

L3   ANSWER 1 OF 1  REGISTRY  COPYRIGHT 2003 ACS
RN   3376-24-7  REGISTRY
CN   2-Propanamine, 2-methyl-N-(phenylmethylene)-, N-oxide (9CI)  (CA INDEX
     NAME)
OTHER CA INDEX NAMES:
CN   Nitrone, N-tert-butyl-.alpha.-phenyl- (6CI, 7CI, 8CI)
OTHER NAMES:
CN   .alpha.-Phenyl-N-tert-butylnitrone
CN   .alpha.-Phenyl-tertbutyl nitrone
CN   2-Methyl-N-(phenylmethylene)-2-propanamine N-oxide
CN   2-Phenyl-N-tert-butylnitrone
CN   Benzylidene-tert-butylamine N-oxide
CN   Benzylidene-tert-butylamine oxide
CN   C-Phenyl-N-tert-butylnitrone
CN   C-Phenyl-N-tert-butylnitrone
CN   N-Benzylidene-tert-butylamine N-oxide
CN   N-Benzylidene-tert-butylamine oxide
CN   N-tert-Butyl-.alpha.-phenylnitrone
CN   N-tert-Butyl-2-phenylnitrone
CN   N-tert-Butyl-C-phenylnitrone
CN   PBN
CN   PBN (amine oxide)
CN   tert-Butyl(benzylidene)amine N-oxide
FS   3D CONCORD
DR   165047-88-1, 173777-90-7, 50643-08-8, 68315-30-0, 154345-12-7, 115995-20-5
MF   C11 H15 N O
CI   COM
LC   STN Files:   AGRICOLA, BEILSTEIN*, BIOBUSINESS, BIOSIS, BIOTECHNO, CA,
       CANCERLIT, CAOLD, CAPLUS, CASREACT, CHEMCATS, CHEMINFORMRX, CHEMLIST,
       CSCHEM, CSNB, DDFU, DETHERM*, DRUGU, EMBASE, IPA, MEDLINE, MRCK*,
       MSDS-OHS, NIOSHTIC, RTECS*, SPECINFO, TOXCENTER, USPAT2, USPATFULL
         (*File contains numerically searchable property data)
     Other Sources:   EINECS**, NDSL**, TSCA**
         (**Enter CHEMLIST File for up-to-date regulatory information)
```



```
**PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT**

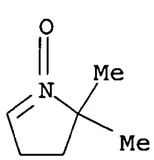
           1011 REFERENCES IN FILE CA (1957 TO DATE)
             24 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
           1010 REFERENCES IN FILE CAPLUS (1957 TO DATE)
              8 REFERENCES IN FILE CAOLD (PRIOR TO 1967)


=> e 3317-61-1/rn
E1             1      3317-59-7/RN
E2             1      3317-60-0/RN
E3             1 --> 3317-61-1/RN
E4             1      3317-62-2/RN
E5             1      3317-63-3/RN
E6             1      3317-66-6/RN
E7             1      3317-67-7/RN
E8             1      3317-68-8/RN
E9             1      3317-72-4/RN
E10            1      3317-75-7/RN
E11            1      3317-77-9/RN
E12            1      3317-78-0/RN

=> s e3
L4             1 3317-61-1/RN

=> d l4

L4   ANSWER 1 OF 1  REGISTRY  COPYRIGHT 2003 ACS
RN   3317-61-1  REGISTRY
CN   2H-Pyrrole, 3,4-dihydro-2,2-dimethyl-, 1-oxide (9CI)  (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN   1-Pyrroline, 5,5-dimethyl-, 1-oxide (6CI, 7CI, 8CI)
OTHER NAMES:
CN   2,2-Dimethyl-3,4-dihydro-2H-pyrrole N-oxide
CN   5,5-Dimethyl-.DELTA.1-pyrroline 1-oxide
CN   5,5-Dimethyl-.DELTA.1-pyrroline N-oxide
CN   5,5-Dimethyl-1-pyrroline 1-oxide
CN   5,5-Dimethyl-1-pyrroline N-oxide
CN   5,5-Dimethyl-4,5-dihydro-3H-pyrrole N-oxide
CN   DMPO
FS   3D CONCORD
MF   C6 H11 N O
CI   COM
LC   STN Files:   AGRICOLA, ANABSTR, BEILSTEIN*, BIOBUSINESS, BIOSIS,
       BIOTECHNO, CA, CANCERLIT, CAOLD, CAPLUS, CASREACT, CHEMCATS,
       CHEMINFORMRX, CHEMLIST, CSCHEM, EMBASE, IFICDB, IFIPAT, IFIUDB, MEDLINE,
       MRCK*, MSDS-OHS, PIRA, TOXCENTER, USPAT2, USPATFULL
         (*File contains numerically searchable property data)
     Other Sources:   EINECS**
         (**Enter CHEMLIST File for up-to-date regulatory information)
```




```
**PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT**

            825 REFERENCES IN FILE CA (1957 TO DATE)
```

```
              46 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
             826 REFERENCES IN FILE CAPLUS (1957 TO DATE)
               7 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

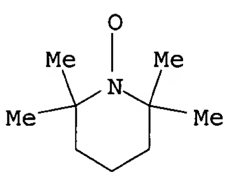

=> e 2564-83-2/rn
E1             1      2564-81-0/RN
E2             1      2564-82-1/RN
E3             1 --> 2564-83-2/RN
E4             1      2564-84-3/RN
E5             1      2564-86-5/RN
E6             1      2564-87-6/RN
E7             1      2564-88-7/RN
E8             1      2564-89-8/RN
E9             1      2564-92-3/RN
E10            1      2564-94-5/RN
E11            1      2564-95-6/RN
E12            1      25640-01-1/RN

=> s e3
L5             1 2564-83-2/RN

=> d l5

L5   ANSWER 1 OF 1  REGISTRY  COPYRIGHT 2003 ACS
RN   2564-83-2  REGISTRY
CN   1-Piperidinyloxy, 2,2,6,6-tetramethyl- (9CI)  (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN   Piperidinooxy, 2,2,6,6-tetramethyl- (7CI, 8CI)
OTHER NAMES:
CN   1,1,5,5-Tetramethylpentamethylene nitroxide
CN   1-Oxyl-2,2,6,6-tetramethylpiperidine
CN   2,2,6,6-Tetramethyl-1-oxylpiperidine
CN   2,2,6,6-Tetramethyl-1-piperadoxyl
CN   2,2,6,6-Tetramethyl-1-piperidinoxyl
CN   2,2,6,6-Tetramethyl-1-piperidinyloxy
CN   2,2,6,6-Tetramethyl-1-piperidyloxy
CN   2,2,6,6-Tetramethylpiperidin-1-oxy
CN   2,2,6,6-Tetramethylpiperidin-1-oxyl radical
CN   2,2,6,6-Tetramethylpiperidin-N-oxyl
CN   2,2,6,6-Tetramethylpiperidine N-oxide
CN   2,2,6,6-Tetramethylpiperidine N-oxide radical
CN   2,2,6,6-Tetramethylpiperidine N-oxy
CN   2,2,6,6-Tetramethylpiperidine N-oxyl
CN   2,2,6,6-Tetramethylpiperidine N-oxyl radical
CN   2,2,6,6-Tetramethylpiperidine nitroxide
CN   2,2,6,6-Tetramethylpiperidine nitroxide radical
CN   2,2,6,6-Tetramethylpiperidine oxide
CN   2,2,6,6-Tetramethylpiperidine-1-oxyl
CN   2,2,6,6-Tetramethylpiperidino-1-oxy
CN   2,2,6,6-Tetramethylpiperidinooxy
CN   2,2,6,6-Tetramethylpiperidinooxy radical
CN   2,2,6,6-Tetramethylpiperidinooxyl
CN   2,2,6,6-Tetramethylpiperidinoxyl
CN   2,2,6,6-Tetramethylpiperidinoxyl radical
CN   2,2,6,6-Tetramethylpiperidinyl 1-oxide
CN   2,2,6,6-Tetramethylpiperidinyl-1-oxyl
CN   2,2,6,6-Tetramethylpiperidinyl-N-oxy
CN   2,2,6,6-Tetramethylpiperidinyloxy
CN   2,2,6,6-Tetramethylpiperidoxyl
CN   HO 6
CN   Tanan
CN   Tanane
CN   Tempo
```

```
CN   TEMPO
CN   TMPO
DR   126517-51-9, 54637-06-8, 125012-91-1, 64104-42-3, 25657-03-8, 26933-82-4
MF   C9 H18 N O
CI   COM
LC   STN Files:   AGRICOLA, ANABSTR, BEILSTEIN*, BIOBUSINESS, BIOSIS, CA,
       CANCERLIT, CAOLD, CAPLUS, CASREACT, CEN, CHEMCATS, CHEMINFORMRX,
       CHEMLIST, CIN, CSCHEM, DDFU, DRUGU, GMELIN*, IFICDB, IFIPAT, IFIUDB,
       IPA, MEDLINE, MRCK*, NIOSHTIC, PIRA, PROMT, RTECS*, TOXCENTER, USPAT2,
       USPATFULL
         (*File contains numerically searchable property data)
     Other Sources:   EINECS**, TSCA**
         (**Enter CHEMLIST File for up-to-date regulatory information)
```



```
            2371 REFERENCES IN FILE CA (1957 TO DATE)
             100 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
            2383 REFERENCES IN FILE CAPLUS (1957 TO DATE)
              23 REFERENCES IN FILE CAOLD (PRIOR TO 1967)


=>

=>  FIL REGISTRY

COST IN U.S. DOLLARS                                   SINCE FILE      TOTAL
                                                            ENTRY    SESSION
FULL ESTIMATED COST                                         13.34      13.55

FILE 'REGISTRY' ENTERED AT 11:15:40 ON 03 MAY 2003
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STRUCTURE FILE UPDATES:     2 MAY 2003   HIGHEST RN 509953-09-7
DICTIONARY FILE UPDATES:    2 MAY 2003   HIGHEST RN 509953-09-7

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 6, 2003

  Please note that search-term pricing does apply when
  conducting SmartSELECT searches.

Crossover limits have been increased.  See HELP CROSSOVER for details.

Experimental and calculated property data are now available.  See HELP
PROPERTIES for more information.  See STNote 27, Searching Properties
in the CAS Registry File, for complete details:
http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf

=>  SET TERMSET E#

SET COMMAND COMPLETED
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```
=>  DEL SEL Y

=>  SEL L1 1 RN

E1 THROUGH E1 ASSIGNED

=>  S E1/RN

L6             1 66893-81-0/RN

=>  SET TERMSET LOGIN

SET COMMAND COMPLETED

=>  FIL CHEMLIST

COST IN U.S. DOLLARS                              SINCE FILE      TOTAL
                                                       ENTRY    SESSION
FULL ESTIMATED COST                                     0.48      14.03

FILE 'CHEMLIST' ENTERED AT 11:15:45 ON 03 MAY 2003
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  FILE COVERS 1979 TO 2 MAY 2003 (20030502/UP)

 This file contains CAS Registry Numbers for easy and accurate substance
 identification.

 TSCA Inventory Tape information is current through January 6, 2003 as
 provided by the January 2003 version from NTIS.

 Updates to the Philippines Inventory of Chemicals and Chemical Substances
 are now available.  The updates are current through December 2002.

 Updates to the Taiwan Toxic Chemical Substances List are now available.
 The updates are curent through July 2002.

 New additions to the file:
  - High Production Volume (HPV) chemicals lists of Australia, ICCA,
    ECD and the United States (HELP HPV)
  - Screening Information Data Set (SIDS) list (HELP SIDS)
  - Substances that are classified as CERCLA Hazardous Substances in
    the Code of Federal Regulations (HELP CERHS)
  - Known health hazards of specific chemicals (HELP HHAZ)
  - Chemical and physical property data (HELP PRP)
  - Information on storage, spill disposal, and environmental fate
    (HELP STOR)
  - International information that may be required for the
    transportation, packaging, and labeling of chemicals (HELP TPL)
  - German Water Hazard Class Substance List (HELP WGK).
  - State of Louisiana Right-to-Know List of Extremely Hazardous
    Substances (HELP SLA)
  - State of Minnesota Right-to-Know Hazardous Substances List
    (HELP SMN)
  - Inventory Update Rule lists for 1986, 1990, 1994, and 1998 (HELP IUR)
  - Violations to Miscellaneous Regulations or Advisory Lists (HELP VIO)

=>  S L6

L7             1 L6
```

```
=> d his

     (FILE 'HOME' ENTERED AT 11:11:52 ON 03 MAY 2003)

     FILE 'REGISTRY' ENTERED AT 11:12:06 ON 03 MAY 2003
                E 66893-81-0/CN
                E POBN/CN
L1            1 S E3
                E 66893-81-0/RN
L2            1 S E3
                E 3376-24-7/RN
L3            1 S E3
                E 3317-61-1/RN
L4            1 S E3
                E 2564-83-2/RN
L5            1 S E3

     FILE 'REGISTRY' ENTERED AT 11:15:40 ON 03 MAY 2003
                SET TERMSET E#
                DEL SEL Y
                SEL L1 1 RN
L6            1 S E1/RN
                SET TERMSET LOGIN

     FILE 'CHEMLIST' ENTERED AT 11:15:45 ON 03 MAY 2003
L7            1 S L6

=> file ca
COST IN U.S. DOLLARS                              SINCE FILE      TOTAL
                                                       ENTRY    SESSION
FULL ESTIMATED COST                                     4.69      18.72

FILE 'CA' ENTERED AT 11:20:02 ON 03 MAY 2003
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FILE COVERS 1907 - 1 May 2003 VOL 138 ISS 19
FILE LAST UPDATED: 1 May 2003  (20030501/ED)

  This file contains CAS Registry Numbers for easy and accurate
  substance identification.


=> s l1
L8          144 L1

=> d l8 139-144 bib,ab

L8   ANSWER 139 OF 144  CA  COPYRIGHT 2003 ACS
AN   92:102211  CA
TI   Photolysis of alkylcobaloximes, methylsalen, cobalamines and coenzyme B12
     in protic solvents:  an ESR and spin-trapping technique study
```

AU Maillard, Ph.; Giannotti, C.
CS Inst. Chim. Subst. Nat., CNRS, Gif sur Yvette, 91190, Fr.
SO Journal of Organometallic Chemistry (1979), 182(2), 225-37
CODEN: JORCAI; ISSN: 0022-328X
DT Journal
LA English
AB The photolysis in protic solvents in the visible region >420 nm of several alkylcobaloximes, cobalamines, methylsalen, and coenzyme B12 was studied by ESR spectroscopy including the use of spin-trapping techniques involving 5,5'-dimethylpyrroline-N-oxide (DMPO), phenyl-N-tert-butylnitrone, and .alpha.-4-pyridyl-1-oxide-N-tert-butylnitrone. During the photolysis, H atoms are probably abstracted from the C(10) position of the corrin equatorial ligand in the case of coenzyme B12 and cobalamine derivs. The ESR spectra of the anaerobic photolysis of alkylcobaloximes in aprotic solvents in the presence of DMPO, followed by the addn. of air or pure O, provide evidence for intraligand radical spin-trapping reactions.

L8 ANSWER 140 OF 144 CA COPYRIGHT 2003 ACS
AN 92:31909 CA
TI Spin trapping and electron spin resonance detection of radical intermediates in the photodecomposition of water at titanium dioxide particulate systems
AU Jaeger, Calvin D.; Bard, Allen J.
CS Dep. Chem., Univ. Texas, Austin, TX, 78712, USA
SO Journal of Physical Chemistry (1979), 83(24), 3146-52
CODEN: JPCHAX; ISSN: 0022-3654
DT Journal
LA English
AB Spin trapping and ESR detection [with the traps .alpha.-Ph N-tert-Bu nitrone and .alpha.-(4-pyridyl N-oxide) N-tert-Bu nitrone] were used to detect free-radical intermediates formed during in situ irradn. of TiO2 and platinized TiO2 powders in aq. solns. Evidence for the prodn. of OH radical (formed in the oxidn. of H2O) and HO2 radical (probably formed in a redn. step) is presented. Other spin adducts, attributed to decompn. reactions of the spin traps, were also found. The results suggest an important role for photogenerated OH in many photocatalytic and photosynthetic processes using TiO2 powders.

L8 ANSWER 141 OF 144 CA COPYRIGHT 2003 ACS
AN 92:2794 CA
TI Spin trapping of superoxide
AU Finkelstein, Eli; Rosen, Gerald M.; Rauckman, Elmer J.; Paxton, John
CS Med. Cent., Duke Univ., Durham, NC, 27710, USA
SO Molecular Pharmacology (1979), 16(2), 676-85
CODEN: MOPMA3; ISSN: 0026-895X
DT Journal
LA English
AB Due to conflicting reports in the literature, the spin trapping of superoxide by the nitrone 5,5-dimethyl-1-pyrroline N-oxide (I) was reinvestigated. Superoxide could indeed be trapped and the 5,5-dimethyl-2-hydroxylpyrrolidinoxyl (II) and 5,5-dimethyl-2-hydroperoxypyrrolidinoxyl (III) adducts were distinguishable. In contrast to earlier work, III was highly unstable and decompd. into II. 2,5,5-Trimethyl-1-pyrroline N-oxide (TMPO), a spin trap structurally similar to I but lacking a .beta.-hydrogen, formed a stable nitroxide upon reaction with superoxide. Rate consts. for the reaction of superoxide with I and TMPO were measured and indicated that spin trapping was an extremely inefficient method for the detection of superoxide. The new spin trap, .alpha.-4-pyridyl-1-oxide-N-tert-butyl nitrone (POBN), was also capable of distinguishing the difference between superoxide and hydroxyl radical. The nitroxide formed upon the reaction of superoxide with POBN was unstable.

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L8   ANSWER 142 OF 144  CA  COPYRIGHT 2003 ACS
AN   91:108398  CA
TI   The ESR spin trapping chemistry of .alpha.-(4-pyridyl 1-oxide)
     N-tert-butyl nitrone and poly(phenyl-N-tert-butyl nitrone)
AU   Wang, Yeeyang
CS   Univ. Georgia, Athens, GA, USA
SO   (1978) 194 pp.  Avail.: Univ. Microfilms Int., Order No. 7914065
     From: Diss. Abstr. Int. B 1979, 39(12, Pt. 1), 5961
DT   Dissertation
LA   English
AB   Unavailable

L8   ANSWER 143 OF 144  CA  COPYRIGHT 2003 ACS
AN   90:5442  CA
TI   On spin trapping hydroxyl and hydroperoxy radicals
AU   Janzen, Edward G.; Nutter, Dale E., Jr.; Davis, Edward R.; Blackburn,
     Barry J.; Poyer, J. Lee; McCay, Paul B.
CS   Guelph-Waterloo Cent. Grad. Work Chem., Univ. Guelph, Guelph, ON, Can.
SO   Canadian Journal of Chemistry (1978), 56(17), 2237-42
     CODEN: CJCHAG; ISSN: 0008-4042
DT   Journal
LA   English
AB   The assignments of the HO.bul., HOO.bul., and ROO.bul. (R = alkyl) radical
     spin adducts of PhCH:N+(O-)CMe3 are discussed.  Results of new expts.
     designed to test the assignments of the first two are described.  The
     HO.bul. adduct assignment is correct.  The relation between a.beta.Hand aN
     is detd. and gives a good fit for the 7 pairs of consts. reported for the
     HO.bul. adduct.  The assignments for the HOO.bul. adduct may be correct
     but no proof is available.

L8   ANSWER 144 OF 144  CA  COPYRIGHT 2003 ACS
AN   89:23427  CA
TI   Spin trapping with .alpha.-pyridyl 1-oxide N-tert-butyl nitrones in
     aqueous solutions.  A unique electron spin resonance spectrum for the
     hydroxyl radical adduct
AU   Janzen, Edward G.; Wang, Y. Y.; Shetty, Raghav V.
CS   Guelph Waterloo Cent. Grad. Work Chem., Univ. Guelph, Guelph, ON, Can.
SO   Journal of the American Chemical Society (1978), 100(9), 2923-5
     CODEN: JACSAT; ISSN: 0002-7863
DT   Journal
LA   English
AB   The spin-trapping chem. of .alpha.-4-pyridyl 1-oxide N-tert-Bu nitrones is
     described in aq. soln. as applied to the problem of detecting HO.bul.
     radical.  Hyperfine-splitting consts. of the hydroxyl adduct are shown as
     a function of pH and H2O2 concn.  A 2nd set of doublets was obsd. which
     were attributed to a 2nd nitroxide; this may be the hydroperoxy adduct,
     but this assignment is unsupported at this time.


=> s l2
L9         144 L2

=> s l3
L10       1014 L3

=> d l10 1009-1014 bib,ab

L10  ANSWER 1009 OF 1014  CA  COPYRIGHT 2003 ACS
AN   63:44176  CA
OREF 63:7904d-e
TI   Derivatives of oxime. VIII. Polarographic reduction of O- and
     N-substituted oximes
AU   Zuman, P.; Exner, O.
CS   Ceskoslov. Akad. Ved, Prague
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SO Collection Czech. Chem. Commun. (1965), 30(6), 1832-52
DT Journal
LA English
AB cf. CA 59, 15203h. In polarography with a dropping Hg electrode, aromatic N-substituted oximes (nitrones) (I) were reduced in acidic as well as in alk. media in a 4-electron wave to the secondary amines. The redn. proceeded through the corresponding Schiff base. The behavior of aromatic O-alkyl derivs. (II) in acidic solns. was similar to that of I whereas in alk. solns. II did not show any redn. wave. Aliphatic N-derivs. were reduced in acidic solns. only, and aliphatic O-derivs. were polarographically inactive. In all instances, both isomeric derivs. could be safely distinguished by polarography in alk. media. 28 references.

L10 ANSWER 1010 OF 1014 CA COPYRIGHT 2003 ACS
AN 54:110179 CA
OREF 54:20925d-i,20926a-i
TI Ozonation of carbon-nitrogen double bonds. I. Nucleophilic attack of ozone
AU Riebel, Alexander H.; Erickson, Ronald E.; Abshire, Claude J.; Bailey, Philip S.
CS Univ. of Texas, Austin
SO J. Am. Chem. Soc. (1960), 82, 1801-7
CODEN: JACSAT; ISSN: 0002-7863
DT Journal
LA Unavailable
AB C-N double bonds of Schiff bases and nitrones were attacked by nucleophilic O3. Thus, N-benzylidene-tert-butylamine (I) yielded the corresponding amide and oxazirane as noncleavage products, and BzH or BzOH and NH4 type salts or N contg. materials as cleavage products. Cleavage was the major reaction with N-benzylideneaniline and N-cyclohexylideneisobutylamine. With N-phenylbenzaldoxime and N-tert-butylbenzaldoxime, O3 gave initially BzH and the corresponding nitroso compds. The latter were oxidized by O3 to nitro compds. O3 was passed into 5 g. I in 130 ml. anhyd. AcOEt, the mixt. cooled to 0.degree. until 1.5 mole equivs. O3 was absorbed, and the mixt. evapd. in vacuo at room temp. A slightly peroxidic powder (0.6 g.) pptd. on addn. of anhyd. Et2O to the residue. An aq. soln. of the solid gave BzOH on addn. of HCl and tert-Bu-NH2 on treatment with NaOH. The amine gave phenyltert-butylthiourea (m. 123-4.degree.) with PhNCO. Evapn. of the Et2O soln. (after filtration of the powder) and crystn. of the residue from petr. ether gave 18% N-tert-butylbenzamide (II), m. 134-5.degree.. Distn. of the filtrate gave 0.5 g. BzH, 0.2 g. I, and 0.5 g. 2-tert-butyl-3-phenyloxazirane (III), b1 68-72.degree., identified by conversion to II. In a similar reaction, 2 mole equivs. O3 was absorbed, the mixt. evapd. in vacuo to half vol., 100 ml. Et2O added, the soln. extd. with 20% NaOH, washed, dried, and evapd. to give 24% II and 15% III. Acidification of the NaOH ext. gave 40% BzOH. When ozonized with O3He the yields were 23% II, 12% III, and 21% BzOH. I with O3 in CH2Cl2 gave 26% II, 5% tert-butylammonium benzoate (IV), sublimed at 120.degree., 9% III, and 28% tert-butylammonium chloride (V). Ozonation with O3He gave 24% V, 20% II, and 8% III. I with O in CH2Cl2 gave no reaction. Following ozonolysis with 1, 1.5, and 2 mole equivs. O3, 40, 9 and 0% I remained as shown by comparison of infrared bands at 908 and 962 cm.-1 I (5 g.) in 130 CCl4 with O3 gave tert-butylamine, 14% II, 0.3 g. IV, 10% III, and a recovery of 10% I. With 2 mole equivs. O3 followed by NaOH treatment as in the AcOEt expts., the yields were 21% II, 9% III, and 32% BzOH. Dibenzoyl peroxide (50 mg.), m. 107-8.degree., was isolated from the distn. residue on trituration with MeOH at -78.degree.. III (3.8 g.) in 75 ml. CH2Cl2 at 0.degree. with O3 oxidized very slowly, 95% O3 (1 mole equiv.) was recovered in a KI trap. The infrared spectrum of the mixt. showed essentially pure III with only very weak CO peak at 1700 cm.-1 (BzH). III in CH2Cl2 with 20% NaOH brought about no change in III as shown by infrared spectra and nothing was obtained from the NaOH layer on acidification. Similar treatment of N-tert-butylbenzaldoxime (VI) in CH2Cl2 with 20% NaOH gave quant. recovery of VI. Similar treatment of I

gave no change. Ozonation of 10 g. N-cyclohexylidene isobutylamine (VII), prepd. in 79% yield by refluxing 1 mole cyclohexanone, 2 moles iso-BuNH2, and 250 ml. C6H6 3 days with removal of H2O gave 48% cyclohexanone and 0.5 g. peroxidic material, believed to be 2-isobutyl-3,3-pentamethyleneoxazirane, b1.5 55-60.degree.. When an aliquot of the mixt. was poured into NaI soln. the iodine released was treated with thiosulfate, which indicated 23% active O material, assumed to be largely oxazirane. Treatment of the remainder of the mixt. as previously described gave 0.2 g. amine salt, assumed to be isobutylammonium chloride. N-Benzylideneaniline (VIII) (5 g.) with O3 in 60 ml. glacial AcOH at 17.degree. absorbed 2 moles O3 and gave 0.33 g. mixt. of PhNO2 and BzH, 39% benzoic acid, and 1.7 g. powdery solid, insol. in Et2O, but sol. in AcOH, mineral acids, MeOH, and C5H5N. The solid with Zn dust gave aniline. VIII treated with NaI and titrated with Na2S2O3 indicated 16% active O. VIII (5 g.) in 100 ml. anhyd. AcOEt at 0.degree. on absorption of 2 moles O3 gave 45% BzOH and 0.35 g. mixt. of PhNO2 and BzH. VIII (5 g.) in anhyd. CH2Cl2 with 1 and 2 mole equivs. O3 at 0.degree. showed considerable amts. of BzH and small amts. of PhNO2. Cryst. material (5 g.) similar to that obtained by ozonation in other solvents was obtained on absorption of 2 equivs. O3 followed by cooling to -78.degree.. With MeOH solvent, benzaldehyde was isolated as the phenylhydrazone (54%). When the mixt. was not reduced with NaI but allowed to reach room temp., the yield of benzaldehyde phenylhydrazone was 24%. Similar results were obtained with an O3-N mixt. N-Benzylidene-m-nitroaniline treated with O3 and the cold mixt. reduced with NaI gave benzaldehyde as the phenylhydrazone (40%). N-Phenylbenzaldoxime (IX) (5 g.) in 75 ml. CH2Cl2 with 2 mole equivs. O3 at -78.degree. developed a green color characteristic of PhNO which disappeared toward the end of the reaction. After evapn. of the solvent in vacuo, the residue was treated with 4 g. semicarbazide-HCl, 40 g. AcONa, 50 ml. H2O, and 50 ml. Skellysolve B, the mixt. shaken, kept cold several hrs., and then filtered to yield 90% benzaldehyde semicarbazone. The org. layer on drying and distg. gave 66% PhNO2. In some runs low yields of BzOH were obtained from partial autoxidn. of BzH. With O3-N, 6.2 g. IX in 70 ml. CH2Cl2 gave 51% BzH and 84% PhNO2, as indicated by infrared analysis. With one mole equiv. O3 (O3-O mixt.), 43% BzH and 30% PhNO were obtained. The reaction repeated with O3 (O3-He mixt.) gave a PhNO fraction which was not isolated, 52% benzaldehyde semicarbazone, and 27% IX. The infrared spectra of BzH, PhNO, PhNO2, BzOH, and IX were detd. at a no. of known concns. and Beer's law plots of various bands obtained. While 6 g. IX was treated with 2 mole equivs. O3 in CH2Cl2 at 0.degree., 10 1-ml. aliquots were withdrawn and their infrared spectra obtained, the concn. of BzH, PhNO, PhNO2, and BzOH being detd. from Beer's plots and found to be 65, 35, 27, and 0%, resp., for 1 mole equiv. and 68, 0, 82, and 15% after 2 mole equivs. O3 was absorbed. Absorption of 2 moles O3 (O3-He mixt.) gave 94% BzH and 98% PhNO2. The yields obtained on gas chromatography of the products through bis(2-ethylhexyl) sebacate and diisodecyl phthalate columns at 163.degree. were 49% BzH, 20% PhNO, and 45% PhNO2 for 1 mole equiv. O3 and 100, 0, and 100% for 2 mole equivs. O3 absorption. PhNO (5.1 g.) in 50 ml. of CH2Cl2 at 0.degree. was completely ozonized with O3-O; 1 mole equiv. O3 was absorbed, the infrared spectra showing only PhNO2. Distn. in vacuo gave 87% PhNO2. VI (5 g.) in 40 ml. CH2Cl2 at -78.degree. with O3 (O3-O mixt.) gave Me3CNO2 (X), b. 120-5.degree., 54% BzH, and 21% BzOH. In a 2nd run the BzH was isolated as the phenylhydrazone (85%). The reaction followed by infrared analysis showed that BzH and X were present in approx. equal quantities until near the end of the reaction, when some autoxidn. of BzH occurred. The corrected yields were 42% BzH, 38% X, and 46% VI after 1 mole equiv. O3 was absorbed, 90% BzH after 1.8 mole equivs. O3, and 46% BzH, 41% BzOH, and 89% X after 2 mole equivs. The course of the reaction was followed by obtaining gas chromatographs of aliquots at 50.degree., 128.degree., and 163.degree. on the same columns as previously. Corrected yields gave 54% BzH, 1% PhNO, and 52% .times. after 1 mole equiv. O3 and 70, 0, and 88% after 2 mole equivs. O3 was absorbed.

L10 ANSWER 1011 OF 1014 CA COPYRIGHT 2003 ACS
AN 52:97888 CA
OREF 52:17226g-i,17227a
TI Preparation of oxaziranes by irradiation of nitrones
AU Splitter, J. S.; Calvin, M.
CS Univ. of California, Berkeley
SO J. Org. Chem. (1958), 23, 651
CODEN: JOCEAH; ~~ISSN:~~ 0022-3263
DT Journal
LA Unavailable
AB .alpha.-(p-Nitrophenyl)-N-ethylnitrone (I), .alpha.-(p-nitrophenyl)-N-tert-butylnitrone (II), and .alpha.-phenyl-N-tert-butylnitrone (III) apparently formed the corresponding oxazirane (IV) (V) (VI), when irradiated. The results were as follows (nitrone, mg. of nitrone, solvent, ml. of solvent, irradiation time, formed, % oxazirane, mode of detn. of yield given): I, 10, MeCN, 70, 1 hr., IV, 35, isolated; II, 10, alc., 70, 25 min., V, 40, isolated; III, 10, MeCN, 50, 2 hrs., VI, 95, from active O content and reconversion to III. The oxazirane structure for IV, V, and VI was established by comparison in each case with the oxazirane obtained by oxidation of the corresponding imine. The properties used for identification of each are as follows: IV, ultraviolet and infrared spectra; V, m. 58-60.degree., and mixed m.p., ultraviolet spectrum, 95% active O; VI, ultraviolet spectrum, almost quant. isomerization to the nitrone III, 90% active O. Although .alpha.-(p-dimethylaminophenyl)-N-phenyloxazirane could not be synthesized by the oxidation of the imine, the properties and reactions of the products of the irradiation of several N, .alpha.-diarylnitrones are consistent with the oxazirane structure. Thus it seems evident that the initial product in the irradiation of nitrones is an oxazirane. The oxazirane is in general at a higher energy level than the nitrone. This photochem. reaction constitutes a conversion and storage of electromagnetic energy as chem. energy.

L10 ANSWER 1012 OF 1014 CA COPYRIGHT 2003 ACS
AN 52:40503 CA
OREF 52:7263d-i,7264a-i,7265a-h
TI Preparation and properties of oxaziranes
AU Emmons, Wm. D.
CS Rohm & Haas, Huntsville, AL
SO J. Am. Chem. Soc. (1957), 79, 5739-54
CODEN: JACSAT; ISSN: 0002-7863
DT Journal
LA Unavailable
AB cf. C.A. 51, 7343h. [Throughout this abstr. R = Me3C and R' = tert-C8H17.] The following imines were prepd. by condensation of the appropriate amine with a ketone or aldehyde (b.p./mm. or m.p., and n20D given): CH2:N R, 65.degree./740, 1.4151; CH2:NR', 50-2.degree./13, 1.4381; PhCH:NR, 90-2.degree./11, 1.5211; p-O2NC6H4CH:N R, 73-5.degree. (petr. ether), -; p-O2N C6CH:NR', -(noncrystallizable oil), 1.5430; p-O2NC6H4CH:NCHMe2 (I), 54-5.degree., -; p-O2NC6H4CH:NEt, 75-6.degree., -; iso-PrCH:NR, 51-3.degree./83, 1.4078; iso-PrCH:NBu, 67.degree./68, 1.4151; iso-PrCH:NCHMePh, 68.degree./0.8, 1.4975; iso-BuMeC:NPr, 65.degree./22, 1.4272;(RN:CH2)2(II), 52-3.degree.,-;BuEtCHCH:NBu,87.degree./8, 1.4338; 2-C5H4NCH:NR, 56-8.degree./0.2, 1.5335; PhCH:NR' (III), 100.degree./0.4, 1.5162; iso-PrMeC:NPr, 48.degree./26, 1.4230; EtMeC:NCH2CH:CH2, 94.degree./100, -; Et2C:NEt, 52-4.degree./54, 1.4230; Me2C:NC6H13, 53-5.degree./5.0, 1.4319; iso-PrCH:NCH2CHMe2, 57.degree./64, 1.4097; Et2C:NCHMePh, 64.degree./0.2, 1.5050. CH2Cl2 (100 cc.) treated with stirring with 30.0 cc. 90% H2O2 and 2 drops H2SO4 and then dropwise with cooling during 0.5 hr. with 135 g. Ac2O, stirred 15 min. at 0.degree. and 30 min. at room temp., added dropwise during 0.5 hr. with stirring to 85 g. CH2:NR in 100 cc. CH2Cl2, kept at room temp. overnight, washed, dried, and fractionated gave 46.4 g. 2-tert-butyloxazirane (IV), b75 52-4.degree., n20D 1.4150, contg. 93.8% active O (detd. with KI and AcOH).

PhCH:NR (V) (80.5 g.) in 100 cc. CH2Cl2 treated dropwise with stirring with 15 cc. 90% H2O2, 50 cc. CH2Cl2, and I drop H2SO4 in 67.2 g. Ac2O and worked up after standing overnight yielded 63.1 g. 3-Ph deriv. (VI) of IV, b0.3 61-3.degree., n20D 1.5081, contg. 95.6% active O. CH2Cl2 (100 cc.), 25.3 cc. 90% H2O2, 2 drops H2SO4, and 114 g. Ac2O added dropwise to 71 g. II in 75 cc. CH2Cl2, kept overnight, and worked up gave 40 g. (crude) bis(2-tert-butyloxazirane), m. 53-6.degree. (petr. ether at -78.degree.), which chromatographed on silica gel gave material, m. 82-4.degree. (presumably meso), and a 2nd fraction, m. 42-3.degree. (presumably dl). Similarly were prepd. the following substituted oxaziranes (substituents in 3, 3, and 2-positions, % yield, b.p. or m.p., mm., active O, and n20D given): H, H, R' (VII), 69, 70-2.degree./6, 99.2, 1.4445; Ph, H, R', 67, -, -, 1.5019; p-O2NC6H4, H, iso-Pr, 60, 46-8.degree., 92.0, -; p-O2NC6H4, H, Et, 97, 34-5.degree., 99.3, -; p-O2NC6H4, H, R, 78, 65-6.degree., 99.4, -; iso-Pr, H, R (VIII), 71, 68-70.degree./39, 99.8, 1.4152; iso-Pr, H, Bu (IX), 65, 65-7.degree./10, 91.5, 1.4178; BuEtCH, H, Bu, 83, -, 98.7, 1.4350; iso-Pr, H, CHMePh (X), 80, -, 99.7, 1.4956; iso-Bu, Me, Pr (XI), 73, 61.degree./8, 93.6, 1.4267; p-O2NC6H4, H, R', 66, 54-6.degree., 96.9, -; Me, iso-Pr, Pr, 64, 60.degree./15, 94.7, 1.4222; Bu, H, H, 74, 43.degree./20, 98.1, 1.4178; iso-Pr, H, R', 78, -, 99.6, 1.4385; Me, Et, CH2CH:CH2, 59, 51.degree./6, 91.2, 1.4413; Et, Et, Et (XII), 56, 62.degree./19, 97.7, 1.4225; Me, Me, C6H13, 14, 58.degree./3, 94.7, 1.4278; iso-Pr, H, iso-Bu (XIII), 50, 53.degree./12, 92.0, 1.4150; Et, Et, MePhCH (XIV), 91, -, 90.1, 1.5038; 2-pyridyl, H, R, 75, 68-70.degree./0.4, 96.1, 1.5010. CH2Cl2 (50 cc.), 9.8 cc. 90% H2O2, 1 drop H2SO4, and 44.1 g. Ac2O added with stirring to 45.9 g. N-cyclohexylideneisobutylamine in 50 cc. CH2Cl2 gave 41.1 g. 2-isobutyl-3,3-pentamethyleneoxazirane (XV), b1.5 59-62.degree., n20D 1.4569, contg. 97.2% active O; after 1 month at room temp. the active O had dropped to 32% and a lower aq. layer had sepd.; the org. layer (21 g.) distd. gave 5.1 g. XV, 7.5 g. cyclohexanone, and 3.5 g. yellow liquid, b0.01 68-70.degree., apparently a condensation product of cyclohexanone with 2 moles Me2CHCH:NH. VI (177 g.) added dropwise with stirring and cooling to 100 cc. H2O, 1 l. MeOH, and 60 cc. H2SO4, warmed, stirred 20 hrs. at room temp., poured into 1 l. H2O, extd. with Et2O, and the ext. distd. gave 98.8 g. BzH, b16 75.degree., and left 1.2 g. PhCH:N(O)R (XVI), m. 75.degree.; the original aq. acidic layer treated with 150 g. NaOH in 300 cc. H2O and extd. 3 days with Et2O yielded 73 g. RNHOH (XVII), m. 64-5.degree. (petr. ether), oxidized in air to blue RNO. III (233 g.) stirred 3 days at room temp. with H2SO4 in aq. MeOH gave similarly 86 g. BzH and 120 g. (crude) R'NHOH (XVIII), b0.02 50-3.degree., m. 40-2.degree. (sublimed), oxidized by air to R'NO. XVII (4.5 g.) and 5.3 g. BzH heated at 45.degree., kept 1 hr. at 50-60.degree., and the product isolated with 50 cc. CH2Cl2 gave 5.5 g. XVI, m. 75-6.degree. (petr. ether). VI (8.8 g.) in 100 cc. dry MeCN refluxed 3 days and the resulting nitrone hydrolyzed in the usual manner gave essentially 100% BzH and XVII. XVIII (14.5 g.) and 10.6 g. BzH heated 0.5 hr. on the steam bath and the product isolated with 50 cc. CH2Cl2 gave 15.8 g. PhCH:N(O)R' (XIX), m. 103-4.degree., hydrolyzed with H2SO4 in aq. MeOH to 100% BzH and XVIII. XVII (8.9 g.) and 4.2 g. 30% aq. (CHO)2 shaken 15 min. at room temp. and the product isolated with 100 cc. CH2Cl2 gave 4.7 g. (crude) [RCN(O):CH]2, creamcolored, m. 193-5.degree. (ligroine). p-O2NC6H4CHO (9.1 g.), 8.9 g. XVII, and 100 cc. C6H6 refluxed 10 hrs. under an H2O-separator gave 10.0 g. p-O2NC6H4CH:N(O)R, yellow, m. 134-5.degree. (3:1 Et2O-petr. ether). XVIII (14.5 g.), 15.1 g. p-O2NC6H4CHO, and 125 cc. C6H6 refluxed 20 hrs. under an H2O-separator yielded 12.8 g. p-O2NC6H4CH:N(O)R', m. 119-21.degree. (petr. ether). I refluxed 14 hrs. in 25 cc. PhMe, evapd., and chromatographed on silica gel gave a mixt. of p-O2NC6H4CHO, an unknown material, and 2.7 g. p-O2NC6H4CH:N(O)CHMe2, m. 98-100.degree. (petr. ether). VI (17.7 g.) in 50 cc. Et2O reduced with 3.8 g. LiAlH4 in 200 cc. Et2O gave 14.6 g. V, b0.1 48.degree.. VI (8.9 g.) added dropwise with stirring to 25 g. KI, 100 cc. H2O, 200 cc. EtOH, and 40 cc. AcOH, treated less than 15 min. with NaHSO3, basified, and extd. with Et2O gave 6.5 g. V. XVI (5.6 g.) in 50 cc. Et2O reduced with 1.2 g. LiAlH4 in 200 cc. Et2O yielded 4.3 g.

PhCH2NROH, m. 71-3.degree. (ligroine). XIX (7.5 g.) in 50 cc. Et2O reduced with 1.2 g. LiAlH4 in 200 cc. Et2O and decompd. with HCl gave 4.5 g. PhCH2NR'OH (XX).HCl, m. 172-4.degree. (EtOAc). XX.HCl and NaOH in aq. MeOH gave XX, noncrystallizable oil. VII (15.7 g.) in 50 cc. Et2O reduced with 3.8 g. LiAlH4 in 150 cc. Et2O gave 10.4 g. R'NHMe, b19 56-8.degree., n20D 1.4305; HCl salt, m. 158-9.degree. (EtOAc). R'N:CH2 reduced with LiAlH4 gave 74% R'NHMe. XI (27.6 g.) and 30.4 g. brucine in 80 cc. CH2Cl2 refluxed 16 hrs. and filtered gave 28.5 g. brucine N-oxide, m. 194.degree. (decompn.); the filtrate washed, dried, and distd. gave 8.1 g. (crude) XI which fractionated yielded 4.3 g. XI, b8.0 60.degree., n20D 1.4260, .alpha.24D -2.80.degree. (neat). VI (35.4 g.) in 100 cc. dry C6H6 treated dropwise with stirring and cooling with 28.4 g. Et2O.BF3 in 50 cc. C6H6, kept 1.5 hrs. at room temp., and filtered yielded 40 g. BF3 salt of the unstable isomer (presumably cis) of XVI, m. 80-8.degree. (CH2Cl2 at -80.degree.), converted on recrystn. from hot EtOAc to the stable isomer (presumably trans) of XVI, m. 135-7.degree.. XVI and Et2O.BF3 in Et2O at room temp. gave an essentially quant. yield of trans-XVI. At room temp. cis-XVI underwent isomerization to trans-XVI. 2-Butyloxazirane (1.0 g.) and 5.0 g. 2,4-(O2N)2C6H3NHNH2 in 25 cc. concd. H2SO4, 36 cc. H2O, and 125 cc. EtOH kept overnight gave 4.2 g. mixed 2,4-dinitrophenylhydrazones of CH2O and PrCHO; the aq. filtrate basified and distd., the aq. alc. distillate shaken 1 hr. at room temp. with 1.35 g. PhNCS, and the product isolated with CH2Cl2 yielded 1.2 g. PhNHCSNH2, m. 152-4.degree.. IX (1.4 g.) gave similarly 4.6 g. mixed 2,4-dinitrophenylhydrazones of equal amts. of PrCHO and iso-PrCHO, and 64% PhNHCSNH2. IV (1.0 g.) under the same conditions yielded 4.0 g. 2,4-dinitrophenylhydrazones of equimolar amts. of CH2O and Me2CO, and 60% MeNH2 (isolated as 1.0 g. PhNHCSNHMe, m. 111-12.degree.). X (1.9 g.) gave similarly 4.6 g. mixed 2,4-dinitrophenylhydrazones of equal amts. of AcH and iso-PrCHO; the alk. filtrate steam-distd. gave 0.8 g. PhNH2 (tribromide, m. 120.degree.). X (19.1 g.) added dropwise with cooling and stirring to 8.6 g. KOH in 100 cc. (CH2OH)2, kept 1 hr. at 0.degree., allowed to warm spontaneously to 45.degree., and heated 1 hr. at 60.degree./5.0 mm. gave 4.1 g. iso-PrCHO (2,4-dinitrophenylhydrazone, m. 179-81.degree.); the mixt. poured into 300 cc. H2O, extd. with CH2Cl2, and the ext. worked up yielded 1.5 g. BzCH:CHCHMe2, m. 139-40.degree.; the mother liquor yielded 3.1 g. PhAc, b12.0 83-5.degree.. VI (5.3 g.) added at room temp. with stirring under N to 12.0 g. Fe(NH4)(SO4)2.6H2O (XXI) in 100 cc. H2O and the product isolated after 2 hrs. with CH2Cl2 gave 5.2 g. BzNHR, m. 134.degree.. A similar run with only 1.2 g. XXI gave 5.3 g. (crude) BzNHR; the petr. ether washings from the crude product gave 1.5 g. unchanged VI. VII (4.7 g.) and 1.2 g. XXI gave similarly 4.1 g. HCONHR', b0.5 84.degree., n20D 1.4555. XII (12.9 g.) added with stirring to 8.0 g. XXI in 100 cc. H2O under He while passing the effluent gases through Drierite, Dry Ice-Me2CO, and liquid N traps, the mixt. stirred 36 hrs. at room temp., extd. 3 days with CH2Cl2, and the ext. distd. gave 4.3 g. Et2CO, b. 96-8.degree., and 3.2 g. EtCONHEt, b0.5 65.degree., n20D 1.4369; the cold traps contained C4H10 and 3:1 C2H6-C2H4; the aq. soln. from a duplicate run washed with Et2O, basified, and distd. gave 0.055 mole NH3. XIV (15.7 g.) and 16 g. XXI in 150 cc. H2O stirred 40 hrs. under N at room temp. gave C4H10 and 2:1 C2H6-C2H4, a trace of PhAc, b12.0 85.degree., and 11.2 g. EtCONHCHMePh, b0.05 121.degree., n20D 1.5262, m. 58-60.degree.; the yield of NH3 was 2%. XIII (14.3 g.) and 8.0 g. XXI in 100 cc. H2O stirred 8 hrs. under He gave 1:1 C3H8-C3H6 and 8.5 g. iso-BuNHOCH, b0.1 60.degree., n20D 1.4388. VIII (14.3 g.), 16 g. XXI, and 150 cc. H2O stirred 16 hrs. under He gave 3:1 C3H8-C3H6 and 8.4 g. HCONHR, b0.2 48.degree., n20D 1.4326. VI (34.4 g.) added dropwise during 2 hrs. at 200.degree. to a vertical glass tube packed with glass helices and entrained with 50 cc. N/min. and the effluent gases passed through an ice and Dry Ice-Me2CO trap gave 7.2 g. Me2C:CH2 contaminated with traces of NO, CO2, and RNO; the ice trap contained 27 g. viscous liquid which yielded 8.8 g. PhCH:NOH and 4.1 g. XVI. VII (30.0 g.) pyrolyzed during 2 hrs. at 200.degree. gave 19.7 g. HCONHR', b0.5 82.degree., n20D 1.4552. VII (15 g.) in 50 cc. HCONMe2 refluxed overnight, poured into H2O, and extd. with CH2Cl2 gave 11.2 g.

HCONHR'. XV (20.0 g.) pyrolyzed during 1 hr. at 300.degree. yielded a trace of cyclohexanone and 16.5 g. N-isobutylcaprolactam, b0.7 75.degree., n20D 1.4750. VIII (25.3 g.) pyrolyzed during 2 hrs. at 250.degree. gave 15.9 g. iso-PrCONHR, m. 118-20.degree. (also obtained from iso-PrCOCl and RNH2), and 3.2 g. unchanged VIII. XIII (20.0 g.) pyrolyzed at 300.degree. gave 9.7 g. iso-Bu-(iso-Pr)NOCH (XXII), b3.0 78-80.degree., n20D 1.4409, and 4.9 g. iso-PrCONHCH2CHMe2 (XXIII), m. 43-5.degree., b0.2 85-8.degree.. iso-BuNHCHMe2 (8.0 g.) treated with stirring with 10.5 g. CCl3CHO and kept at room temp. overnight yielded 7.5 g. XXIII, b1.0 65.degree., n20D 1.4411. Iso-PrCOCl (11.2 g.) in 100 cc. dry Et2O treated with stirring with 14.6 g. iso-BuNH2, refluxed 1 hr., and worked up gave 13.0 g. XXIII, m. 43-5.degree., b0.1 82.degree.. XI (100 g.) pyrolyzed during 4 hrs. at 300.degree. yielded 67.0 g. mixt. of 64% iso-BuCONMePr (b5.0 93.degree., n20D 1.4437, from iso-BuCOCl and MeNHPr) and 36% iso-BuPrNAc (b5.0 91.degree., n20D 1.4440, from iso-BuNHPr and Ac2O). XI (25.0 g.) pyrolyzed at 300.degree. yielded 13.9 g. mixt. of 39% iso-PrPrNAc (b5.0 68-70.degree., n20D 1.4437, from iso-PrNHPr and Ac2O) and 61% iso-PrCONMePr (b5.5 67.degree., n20D 1.4409, from iso-PrCOCl and MeNHPr). XI (27.6 g.) refluxed 2 hrs. under N at 168 to 128.degree. and the effluent gases passed over aq. H3BO3 showed the formation of 0.057 mole NH3; the liquid product distd. gave 16.1 g. iso-BuAc, b. 114-16.degree., 0.8 g. unreacted XI, and 1.0 g. mixed amides, b3.0 68-70.degree.. XIII (25.0 g.) refluxed 3 hrs. under N at 165 to 105.degree. yielded 8.0 g. iso-PrCH:NCH2CHMe2, b47 60.degree..

L10 ANSWER 1013 OF 1014 CA COPYRIGHT 2003 ACS
AN 52:35153 CA
OREF 52:6304h-i,6305a-b
TI Kinetics of the thermal isomerization of 2-tert-butyl-3-phenyloxazirane
AU Hawthorne, M. Frederick; Strahm, R. Donald
CS Rohm & Haas Co., Huntsville, AL
SO J. Org. Chem. (1957), 22, 1263-4
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA Unavailable
AB This kinetic study was designed to det. the enthalpy and entropy of activation for the rearrangement of 2-tert-butyl-3-phenyloxazirane (I), in diethylene glycol diethyl ether (II) over a 40.degree. temp. range. I was further purified by chromatography on silica gel using CH2Cl2 followed by vacuum distn. The ultraviolet spectrum of I in MeCN showed only end absorption. When repurified, II b33 94-6.degree., n20D 1.4115. Similarly pure N-tert-butylbenzaldoxime (III) was obtained, m. 75-7.degree., .lambda. 289 m.mu., .epsilon. 16,8000. Solns. of I (0.1 to 0.44M) in pure II were prepd. in 10-ml. flasks, the flasks placed at zero time in the appropriate thermostat, at intervals aliquots were removed and delivered to a vol. of MeCN which was made up to 10 ml., optical density readings were made at 298 m.mu. and the concn. of III computed. Points were taken until the reaction had progressed to 75% completion. After several days (or weeks at lower temp.) the rearrangement of I to III was found to be complete. First order plots were prepd. by plotting log [I] against time. The following first order consts. were obtained (temp., initial I concn. in mole/l., k1 (sec.-1) .times. 107 are given): 100.0.degree., 0.437, 545; 100.degree., 0.252, 537; 85.0.degree., 0.116, 530; 85.0.degree., 0.241, 119; 85.0.degree., 0.229, 113; 85.0.degree., 0.120, 113; 85.0.degree., 0.115, 117; 85.0.degree., 0.233, 5.40; 60.0.degree., 0.127, 5.40. The .DELTA. H was found to be 28 kcal./mole between 60 and 100.degree.. The entropy of activation was -3 .+-. 1 entropy units, a value which indicated that the structures of oxazirane reactant and nitrone product were equally rigid. The slight neg. value may indicate increased solvation of the transition state due to the development of a strong N .fwdarw. O dipole.

L10 ANSWER 1014 OF 1014 CA COPYRIGHT 2003 ACS
AN 51:39192 CA
OREF 51:7343h-i,7344a

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TI   Synthesis of oxaziranes
AU   Emmons, Wm. D.
CS   Redstone Arsenal, Huntsville, AL
SO   J. Am. Chem. Soc. (1956), 78, 6208-9
     CODEN: JACSAT; ISSN: 0002-7863
DT   Journal
LA   Unavailable
AB   Certain azomethines which are sluggish toward acid hydrolysis can be
     oxidized in good yield with (AcO)2 to oxaziranes (I), RR1.C.NR2.O, which
     resemble org. peroxides.  Some typical examples of I are (R, R1, R2,
     b.p./mm., % yield, and nD20 given): H, H, tert-Bu, 52.degree./75, 46,
     1.4150; Ph, H, tert-Bu, 63.degree./0.5, 71, 1.5081; H, H, tert-C8H17,
     64.degree./6, 69, 1.445; p-O2NC6H4, H, Et, - (m. 34-5.degree.), 95, -; Me,
     iso-Bu, Pr, 64.degree./7, 63, 1.4277.  2-tert-Butyl-3-phenyloxazirane (II)
     with H2SO4 in aq. MeOH yielded 100% BzH and Me3CNHOH (III), m. 64.degree..
     III and BzH yielded the nitrone, PhCH:N(O)CMe3 (IV), m. 76.degree..  II
     with LiAlH4 in Et2O yielded only Me3CN:CHPh; IV yielded PhCH2N(OH)CMe3, m.
     72.degree..  2-Propyl-3-methyl-3-isobutyloxazirane was partially resolved
     with brucine in boiling CH2Cl2.


=> s l11
L11 NOT FOUND
The L-number entered has not been defined in this session, or it
has been deleted.  To see the L-numbers currently defined in this
session, enter DISPLAY HISTORY at an arrow prompt (=>).

=> d his

     (FILE 'HOME' ENTERED AT 11:11:52 ON 03 MAY 2003)

     FILE 'REGISTRY' ENTERED AT 11:12:06 ON 03 MAY 2003
                E 66893-81-0/CN
                E POBN/CN
L1            1 S E3
                E 66893-81-0/RN
L2            1 S E3
                E 3376-24-7/RN
L3            1 S E3
                E 3317-61-1/RN
L4            1 S E3
                E 2564-83-2/RN
L5            1 S E3

     FILE 'REGISTRY' ENTERED AT 11:15:40 ON 03 MAY 2003
                SET TERMSET E#
                DEL SEL Y
                SEL L1 1 RN
L6            1 S E1/RN
                SET TERMSET LOGIN

     FILE 'CHEMLIST' ENTERED AT 11:15:45 ON 03 MAY 2003
L7            1 S L6

     FILE 'CA' ENTERED AT 11:20:02 ON 03 MAY 2003
L8          144 S L1
L9          144 S L2
L10        1014 S L3

=> s l2
L11         144 L2

=> s l1
L12         144 L1
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                E 66893-81-0/CN
                E POBN/CN
L1            1 S E3
                E 66893-81-0/RN
L2            1 S E3
                E 3376-24-7/RN
L3            1 S E3
                E 3317-61-1/RN
L4            1 S E3
                E 2564-83-2/RN
L5            1 S E3

     FILE 'REGISTRY' ENTERED AT 11:15:40 ON 03 MAY 2003
                SET TERMSET E#
                DEL SEL Y
                SEL L1 1 RN
L6            1 S E1/RN
                SET TERMSET LOGIN

     FILE 'CHEMLIST' ENTERED AT 11:15:45 ON 03 MAY 2003
L7            1 S L6

     FILE 'CA' ENTERED AT 11:20:02 ON 03 MAY 2003
L8          144 S L1
L9          144 S L2
L10        1014 S L3
L11         144 S L2
L12         144 S L1
L13        1014 S L3

=>  s l4
L14         825 L4

=> d l14 820-825 bib,ab

L14  ANSWER 820 OF 825  CA  COPYRIGHT 2003 ACS
AN   64:75320  CA
OREF 64:14065e
TI   Mass spectra of N-oxides
AU   Grigg, R.; Odell, B. G.
CS   Univ. Chem. Lab., Cambridge, UK
SO   J. Chem. Soc., B, Phys. Org. (1966), (3), 218-19
DT   Journal
LA   English
AB   Large (M-16) ions are not characteristic of the N-oxide groups in
     1-pyrroline N-oxides but are of some diagnostic value in aromatic
     N-oxides.

L14  ANSWER 821 OF 825  CA  COPYRIGHT 2003 ACS
AN   64:51877  CA
OREF 64:9667e-f
TI   The periodate oxidation of nitrones
AU   Qureshi, A. K.; Sklarz, B.
CS   Imp. Coll. Sci. Technol., London
SO   J. Chem. Soc., Org. (1966), (4), 412-15
DT   Journal
LA   English
AB   The periodate oxidn. of some 1-pyrroline 1-oxides with two substituents at
     C-5 but unsubstituted at C-2 leads to a series of 4-nitrosopentanoic
     acids.  Evidence is adduced for the course of the reaction.  An
     N-alkylhydroxy amino compd. is oxidized in chloroform to the nitroso
     compd. by tetraethylammonium periodate, a potentially useful reagent, sol.
     in various org. solvents.
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L14 ANSWER 822 OF 825 CA COPYRIGHT 2003 ACS
AN 62:90756 CA
OREF 62:16185g-h,16186a
TI Oxidative phosphorylation, by phosphate adducts of compounds with a C=N+ double bond
AU Clark, V. M.; Hutchinson, D. W.; Wilson, D. E.
CS Univ. Chem. Lab., Cambridge, UK
SO Angew. Chem. (1965), 77(6), 259-60
DT Journal
LA German
AB I-III, on treatment with (Bu4N)3PO4 or (Me3SO)3PO4 in non-aq. solvents, gave adducts which could be detd. spectroscopically. With 100% excess phosphate, followed by oxidn. with Br or KMnO4, 25% pyrophosphate was formed. The adduct of Ib with an equiv. amt. of phosphate, on reaction with (Bu4N)2PhPO4 and KMnO4, gave 25% monophenyl pyrophosphate. Addn. of Br to a soln. of the adduct of III in EtOH yielded 10% EtH2PO4. A reaction scheme was proposed.

L14 ANSWER 823 OF 825 CA COPYRIGHT 2003 ACS
AN 57:62584 CA
OREF 57:12408d-e
TI Studies in the pyrroline series. I. The proton mag-netic resonance spectra of some pyrrolines
AU Bonnett, R.; McGreer, D. E.
CS Univ. Brit. Columbia, Vancouver
SO Can. J. Chem. (1962), 40, 2177-80
DT Journal
LA Unavailable
AB The proton magnetic spectra of 2,4,4-trimethylpyrroline, 5,5-dimethylpyrroline, 2-methylpyrroline, 2,2,3,3-tetramethylpyrroline, 2-phenyl3,3-dimethylpyrroline, 3,3-dimethylpyrroline N-oxide, and 5,5-dimethylpyrroline N-oxide was measured. The proton magnetic resonance spectra strongly reinforce previous conclusions that the double bond is in the .DELTA.1 position.

L14 ANSWER 824 OF 825 CA COPYRIGHT 2003 ACS
AN 55:33039 CA
OREF 55:6466d-f
TI Cycloaddition of .alpha.,.beta.-unsaturated esters to nitrones
AU Delpierre, G. R.; Lamchen, M.
CS Univ. Cape Town, S. Afr.
SO Proc. Chem. Soc. (1960) 386-7
DT Journal
LA Unavailable
AB EtO2CCH:CH2 added to 5,5-dimethyl-1-pyrroline 1-oxide (I) to give Me2C.CH2.CH2.N.CH.CH2.CH(CO2Et).O. The structure of this product was confirmed by redn. with LiAlH4 in boiling tetrahydrofuran to give 2,2-dimethyl-5-(2,3-dihydroxypropyl)pyrrolidine, reduced with red P and HI followed by Zn and HCl to 2,2-dimethyl-5-propylpyrrolidine (picrolonate m. 218.degree.; oxalate m. 201.degree.), identical with the product of the action of PrMgBr on I followed by redn. Redn. of the product from Me2CHMgBr and I gave 2,2-dimethyl-5-isopropylpyrrolidine; picrolonate decompd. 245.degree.; oxalate m. 221.degree..

L14 ANSWER 825 OF 825 CA COPYRIGHT 2003 ACS
AN 54:2159 CA
OREF 54:485c-i,486a-i,487a-e
TI Experiments towards the synthesis of corrins. II. Preparation and reactions of 1-pyrroline 1-oxides
AU Bonnett, R.; Brown, R. F. C.; Clark, V. M.; Sutherland, I. O.; Todd, Alexander
CS Univ. Cambridge, UK
SO J. Chem. Soc. (1959) 2094-2102
DT Journal

LA Unavailable

AB cf. C.A. 53, 20036h. The reduction of .gamma.-nitro carbonyl compds. to give 1-pyrroline 1-oxides was described and the reactions of several such N-oxides reported. 4,4-Dimethyl-5-nitro-2-pentanone (33 g.) and 9 g. NH4Cl in 250 ml. H2O stirred 2 hrs. with addn. of 45 g. Zn dust, stirring continued a further 2 hrs., the mixt. filtered, the filtrate and washings evapd., the sirup dissolved in 75 ml. CHCl3, and the CHCl3 distd. gave 19.8 g. 2,4,4-trimethyl-1-pyrroline 1-oxide(I), hygroscopic oil, b0.4 72.degree., .nu. 1613 cm.-1 .lambda. 229 m.mu., .epsilon. 9000; picrate m. 111.degree. (alc.); MeI deriv., prisms, m. 106.degree. (decompn.) (alc.-Et2O). Freshly distd. MeCH:CHCHO (17.5 g.) in 25 ml. anhyd. MeOH added dropwise to 25 g. 2-O2NCHMe2 in 40 ml. MeOH contg. NaOMe from 1 g. Na at 60.degree., stirred 3 hrs., left overnight, 3 ml. AcOH added, the MeOH evapd., the residue poured into H2O, the product extd. with Et2O, the ext. evapd., and distd. gave 20.4 g. 3,4-dimethyl-4-nitropentanal (II), b0.6 80.degree.; semicarbazone, leaflets, m. 150-1.degree. (aq. alc.). Reduction of 60 g. II with 74 g. Zn dust and 15 g. NH4Cl in 400 ml. H2O gave 24.3 g. 4,5,5-trimethyl-1-pyrroline 1-oxide (III), b1 85.degree., .lambda. 234 m.mu., .epsilon. 8800, .nu. 1572 cm.-1; picrate, lemon yellow laths, m. 112.degree. (alc.). 2-(3-Methyl-3-nitrobutyl)-1,3-dioxolane (50 g.) and 15 g. NH4Cl in 300 ml. H2O at 10.degree. treated during 20 min. with 70 g. Zn dust, the mixt. stirred 15 min., filtered, the combined filtrate and washings acidified, left overnight, heated 1 hr. at 75.degree., evapd. to 150 ml., made alk., reduced to 100 ml., after satn. with borax, the soln. extd. with CHCl3, dried, and fractionated gave 23.6 g. 5,5-dimethyl-1-pyrroline 1-oxide (IV), b0.6 66-7.degree., .lambda. 234 m.mu., .epsilon. 7700, .nu., 1573 cm.-1; picrate, yellow needles, m. 81.degree.. Reduction of 4-methyl-4-nitropentanal with Zn dust and NH4Cl gave 27% IV and a high boiling residue. 3-(3,4-Methylenedioxyphenyl)-4-nitro-1-phenylbutan-1-one (3.13 g.) and 0.50 g. NH4Cl similarly treated with Zn in tetrahydrofuran and the product isolated as above gave 1.12 g. 4-(3,4-methylenedioxyphenyl)-2-phenyl-1-pyrroline 1-oxide (V), m. 145.degree. (CH2Cl2-Et2O), .nu. 1613 cm.-1 Reduction of 300 mg. V with 1.2 g. Zn dust and 0.3 g. NH4Cl in 30 ml. 60% aq. MeOH 8 hrs. at room temp. gave 4-(3,4-methylenedioxyphenyl)-2-phenyl-1-pyrroline, isolated at the HCl salt (200 mg.), m. 235-40.degree. (decompn.). CH2:CHCHO (14 ml.) added to 36 g. Et .alpha.-nitropropionate in 200 ml. alc. contg. NaOEt (from 0.3 g. Na), the mixt. kept 3 hrs. at 40-50.degree., 1 ml. AcOH added, the solvent evapd., the product extd. with C6H6, and distd. gave 31.5 g. Et 2-methyl-2-nitro-5-oxopentanoate (VI), b0.5 107.degree.; 2,4-dinitrophenylhydrazone, orange needles, m. 75-6.degree. (alc.). VI and (CH2OH)2 in the presence of p-MeC6H4SO3H gave 78% of the 1,3-dioxolane (VII), b0.5 121-5.degree.. VII (28.5 g.) reduced with 41.5 g. Zn dust in 200 ml. 50% aq. alc. contg. 5.5 g. NH4Cl, the soln. concd., extd. with CHCl3, and the soln. evapd. gave 26 g. crude hydroxylamine, b0.3 140-55.degree., viscous oil. The hydroxylamine (25.5 g.) in 400 ml. 0.3N HCl left overnight at room temp., neutralized with NH4OH, evapd. below 60.degree., the residue extd. with CHCl3, and fractionated gave 11.6 g. 5-ethoxycarbonyl-5-methyl-1-pyrroline 1-oxide (VIII), b0.3 120.degree.. Treatment of 2.13 g. VIII 1 hr. at 100.degree. with 20 ml. 2.5% NaOH, passage of the soln. through Dowex 50 (H form), and evapn. of the acidic eluate gave 1.37 g. 5-carboxy-5-methyl-1-pyrroline 1-oxide (VIIIa), m. 135-6.degree. (decompn.) (CHCl3), .lambda. 235-236 m.mu., .epsilon. 7700. KBH4 (0.4 g.) and 2.5 g. I in 10 ml. H2O kept 2 days at room temp., satd. with K2CO3, extd. with Et2O, the ext. evapd., and the residue fractionated gave 0.86 g. of the hydroxylamine (IX), b25 95-100.degree.; H oxalate m. 88.degree. (Me2CO-Et2O). Both IX and the salt readily reduced aq. alk. triphenyltetrazolium chloride (IXa) to the red formazan. IX (0.5 g.) in 10 ml. H2O contg. 1 ml. NH4OH and 5 mg. CuSO4 was reoxidized during 2 hrs. to I by passing air through the soln. III (0.63 g.) reduced as above with KBH4 in H2O and the cyclic hydroxylamine isolated gave the crude base; addn. of alc. HCl gave 1-hydroxy-2,2,3-triphenylpyrrolidine-HCl, prisms, m. 163.degree. (alc.-Et2O). Titration in aq. soln. indicated pKa 5.9. The base readily reduced IXa. I (0.5 g.), 5 g. Zn dust, 2 ml. AcOH, and 8

ml. H2O refluxed 5 hrs., 2 ml. more AcOH added, heating continued a
further 20 hrs., the soln. made alk., and steam distd. gave with picric
acid 0.88 g. 2,4,4-trimethyl-1-pyrrolinium picrate (X), m. 192-3.degree.
(alc.). I 12 hrs. at room temp. in CHCl3 satd. with SO2 gave 15% X. I
(0.5 g.), 4 g. granular Sn, 5 ml. concd. HCl, and 5 ml. H2O refluxed 20
hrs. and worked up as usual gave 0.13 g. X. The mother liquors afforded
0.32 g. 2,4,4-trimethylpyrrolidinium picrate, m. 162.degree..
1-Hydroxy-2,4,4-trimethylpyrrolidine (0.63 g.) and 0.92 g. p-MeC6H4SO2Cl
in 10 ml. Et2O treated with 0.98 g. NEt3 in 5 ml. Et2O gave when left
overnight at room temp. 1.7 g. triethylammonium salts; the filtrate extd.
with dil. HCl, the acid ext. made alk., satd. with K2CO3, extd. with Et2O,
and gas chromatographed indicated that the amine product was
2,4,4-trimethyl-1-pyrroline, identified as X (33% yield).
1-Hydroxy-2,2,3-trimethylpyrrolidine (1.1 g.) and 1.55 g. p-MeC6H4SO2Cl in
15 ml. Et2O treated overnight with 1.64 g. NEt3 in 5 ml. Et2O gave an
ethereal soln. of the crude bases. These transferred to H2O by dil. H2SO4
and the aq. soln. placed on a column of Amberlite IRC-50 gave 1.08 g.
4,5,5-trimethyl-1-pyrrolinium picrate, prisms, m. 172.degree.
(Me2CO-Et2O). IV (5 g.) and 3.9 g. KCN in 20 ml. H2O treated during 1.5
hrs. at 0.degree. with 25 ml. 2N HCl, after a further 2 hrs. the mixt.
brought to pH 11, and extd. gave 4.8 g. 2-cyano-1-hydroxy-5,5-
dimethylpyrrolidine (XI), prisms, m. 92.degree. (Et2O-ligroine), .nu. 3230
and 2240 cm.-1 XI (8.1 g.) and 1.5 g. Cu(OAc)2 in 100 ml. 60% aq. alc.
contg. 5 ml. NH4OH aerated until a blue color was restored, concd. to 30
ml., extd. with CHCl3, and fractionated gave 5.8 g. 2-cyano-5,5-dimethyl-1-
pyrroline 1-oxide (XII), b0.5 110.degree., .nu. 2220 and 1540 cm.-1,
.lambda. 271 m.mu., .epsilon. 10,700. III with HCl and KCN as above gave
66% 2-cyano-1-hydroxy-4,5,5-trimethylpyrrolidine (XIII), m. 109.degree.
(Et2O-ligroine), .nu. 3360, 3290, 2270, and 2240 cm.-1 Cu catalyzed air
oxidn. of XIII gave 76% 2-cyano-4,5,5-trimethyl-1-pyrroline 1-oxide (XIV),
plates, m. 86-7.degree. (EtOAc-ligroine), .nu. 2210 and 1528 cm.-1,
.lambda. 273 m.mu., .epsilon. 11,100. VIIIa (0.80 g.) and 0.37 g. KCN in
10 ml. H2O left 6 hrs. at room temp., the soln. passed through a column of
Dowex 50 resin, and the acidic eluate evapd. gave 0.75 g.
5-cyano-1-hydroxy-2-methylpyrrolidine-2-carboxylic acid, m. 133-4.degree.
(decompn.) (EtOAc), pKa 3.6, .nu. 3250, 2245, and 1698 cm.-1
2,4,4-Trimethyl-1-pyrroline 1-oxide was recovered in 52% yield after
attempted addn. of HCN under the above conditions. XIV (4.1 g.) in 40 ml.
10% aq. NaOH refluxed 3 hrs., the soln. neutralized, and extd. with CHCl3
gave 1.1 g. 1-hydroxy-4,5,5-trimethyl-2-pyrrolidone, needles, m.
101-2.degree. (Et2O-ligroine), .nu. 3310, 3120, and 1680 cm.-1, pKa 8.85,
deep reddish purple color with FeCl3. The original aq. soln. adjusted to
pH 1 and extd. with CHCl3 gave 2.4 g. 2-carboxy-4,5,5-trimethyl-1-
pyrroline 1-oxide (XIVa), m. 40-1.degree. (C6H6), .lambda. 266 m.mu.,
.epsilon. 8200, pKa 2.85. XII (5 g.) hydrolyzed as above, acidified to pH
1, and extd. with CHCl3 gave 3.1 g. 2-carboxy-5,5-dimethyl-1-pyrroline
1-oxide (XIVb), m. 86.degree. (Et2O-ligroine), pKa 2.80, .lambda. 265
m.mu., .epsilon. 8600. The mother liquor from the above crystn. evapd.
and an aq. soln. passed down a column of Dowex 50 resin gave 1 g.
1-hydroxy-5,5-dimethyl-2-pyrrolidone (XV), m. 82-3.degree. (hexane), pKa
8.7, .nu. 3320, 3090, and 1678 cm.-1 An authentic sample of XV prepd. by
reduction of 21 g. Me 4-methyl-4-nitropentanoate with 30 g. Zn dust and 6
g. NH4Cl in 50% aq. alc., the mixt. stirred 4 hrs., the solids removed,
the filtrate evapd. to a sirup, this sirup in 50 ml. 2N HCl extd. with
CHCl3, and evapd. gave 4.8 g. XV. XIVa (100 mg.) was heated 3 min. at
140-50.degree. (CO2 evolved), and the oily residue converted into the
picrate, identified as 120 mg. III picrate. XIVb treated as above gave
XII, isolated as the picrate, m. 78-9.degree.. XII (10 g.) in 30 ml. Et2O
refluxed 0.5 hr. with EtMgBr (from 15 g. EtBr and 3.4 g. Mg), excess aq.
NH4Cl added, the Et2O layer sepd., dried, and fractionated gave 11.2 g.
5-ethyl-1-hydroxy-2,2-dimethylpyrrolidine (XVI), b0.7 50-3.degree.. Air
oxidn. of XVI in aq. alc. contg. Cu(OAc)2 and NH4OH gave 90% corresponding
nitrone, b0.2 54-6.degree., .nu. 1600 cm.-1; picrate, yellow needles, m.
71-2.degree. (alc.). XII (2 g.) treated as above with MeMgI followed by

Cu catalyzed air oxidn. of the intermediary hydroxylamine and treatment with picric acid gave 1.9 g. 2,5,5-trimethyl-1-pyrroline 1-oxide picrate, m. 98.degree.(alc.). I (10 g.) treated with MeMgI gave 9 g. 1-hydroxy-2,2,4,4-tetramethylpyrrolidine (XVII), sublimed at 75.degree./14 mm., m. 62.degree.. Cu catalyzed air oxidation of 8.5 g. XVII gave 72% 3,3,5,5-tetramethyl-1-pyrroline 1-oxide, b1 73.degree., m. 32-4.degree. (ligroine); picrate m. 137-8.degree.(alc.). MeNO2 (11.7 g.) in 50 ml. alc. left at room temp. overnight with 10.5 g. IV and 1.8 g. Na in 200 ml. alc., acidified, and evapd. gave 11.8 g. 1-hydroxy-2,2-dimethyl-5-nitromethylpyrrolidine, yellow oil, b0.3 98.degree., .nu. 3220 and 1552 cm.-1 Similarly, 12.6 g. EtNO2 added to 9.5 g. IV gave 3.7 g. 1-hydroxy-2,2-dimethyl-5-(1-nitroethyl)pyrrolidine, b0.4 100-2.degree., .nu. 3480 and 1548 cm.-1; 5.7 g. IV was recovered. I (0.50 g.), 0.45 g. BzH, and 50 mg. KOH in 4 ml. alc. refluxed 0.5 hr., poured into H2O, and crystd. gave 0.31 g. 4,4-dimethyl-2-styryl-1-pyrroline 1-oxide, m. 113.degree. (C6H6-ligroine), .nu. 1537, 990, 977, 756, and 691 cm.-1, .lambda. 236 and 329 m.mu., .epsilon. 11,200 and 22,600. A similar condensation wtih p-O2NC6H4CHO gave 4,4-dimethyl-2-(4-nitrostyryl)-1-pyrroline 1-oxide, leaflets, m. 233.degree. (alc.). I (0.4 g.) benzoylated and the crude material passed through Al2O3 and concd. gave 0.51 g. 5-benzamido-1-benzoyloxy-4,4-dimethylpentan-2-one (XVIII), prisms, m. 117.degree. (80% MeOH), .nu. 3360, 1718, 1637, and 1547 cm.-1, .lambda. 231 and 270 m.mu., .epsilon. 25,700 and 1800. Hydrolysis of XVIII (113 mg.) with aq. alc. KOH gave 67 mg. BzOH; 2,4-dinitrophenylhydrazone, prisms, m. 172.degree. (alc.), .lambda. 356-8 m.mu., .epsilon. 21,900.

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=> d his

     (FILE 'HOME' ENTERED AT 11:11:52 ON 03 MAY 2003)

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                E POBN/CN
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                E 66893-81-0/RN
L2            1 S E3
                E 3376-24-7/RN
L3            1 S E3
                E 3317-61-1/RN
L4            1 S E3
                E 2564-83-2/RN
L5            1 S E3

     FILE 'REGISTRY' ENTERED AT 11:15:40 ON 03 MAY 2003
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                DEL SEL Y
                SEL L1 1 RN
L6            1 S E1/RN
                SET TERMSET LOGIN

     FILE 'CHEMLIST' ENTERED AT 11:15:45 ON 03 MAY 2003
L7            1 S L6

     FILE 'CA' ENTERED AT 11:20:02 ON 03 MAY 2003
L8          144 S L1
L9          144 S L2
L10        1014 S L3
L11         144 S L2
L12         144 S L1
L13        1014 S L3
L14         825 S L4

=> s l5
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L15 2379 L5

=> d l15 2374-2379 bib,ab

L15 ANSWER 2374 OF 2379 CA
AN 61:54445 CA
OREF 61:9381h,9382a-b
TI Nitroxides. VI. Stable piperidine and pyrrolidine free radicals. Effects of solvents on the ultraviolet and electron paramagnetic resonance spectra. Conformational mobility of radicals
AU Briere, R.; Lemaire, H.; Rassat, A.
CS C. E. N., Grenoble, Fr.
SO Tetrahedron Letters (1964), (27-28), 1775-80
DT Journal
LA French
AB An essential property of the nitroxides I (R = O; H, OH; H2; :NOH; Me, OH) (II, III, IV, V, VI) and VII is the nonbasic character, which permits their sepn. from the starting amine by washing with acid. The nonbasicity indicates a participation of the N free pair in a 3-electron N-O linkage and a shielding of the unpaired electron of O from external attack, thus contributing to the stability of the radicals. The radicals showed ultraviolet (UV) absorption bands at .lambda. 405-450 m.mu. (.epsilon. 24,700-22,222) sensible to solvent effect, decreasing from nonpolar to polar solvents, and a 2nd band around .lambda. 240 m.mu. (.epsilon. 3000). As a 1st approximation, the nitroxide group can be described as a ketyl group, a carbonyl with an electron fixed in the 1st antibonding level .pi.*. The behavior of the band at 450 m.mu., varying with the solvent, is characteristic of a forbidden transition .pi. .fwdarw. .pi.* by reasons of symmetry. The frequencies of max. absorption varies linearly with the Kosower Z factor. Similarly, the variation of aN, the hyperfine splitting of N found in electron spin resonance (e.s.r.), on change of solvent is related to the factor Z and not to the dielec. const. of the medium. Although I and VII have closely related structures, the e.s.r. spectra permit observation of various characteristics related to the structure, in particular the variation of aN with the solvent and with the structure, and in compds. II, V, and VI the variation of a13c with the structure.

L15 ANSWER 2375 OF 2379 CA
AN 60:52651 CA
OREF 60:9240c-h
TI Organic radical reactions involving no free valence
AU Rozantsev, E. G.; Neiman, M. B.
CS Inst. Chem.-Phys., Acad. Sci. U.S.S.R., Moscow
SO Tetrahedron (1964), 20(1), 131-7
DT Journal
LA Unavailable
AB A radical, possessing a reactive group together with the free valence, might react in 2 ways and an attempt was made to direct the chem. process through the reactive group by increasing the steric hindrance close to the atom carrying the free valence, thus leaving aside the unpaired electron. The previously unobserved heterocyclic free radical, 2,2,6,6-tetramethyl-4-piperidone nitrogen oxide (I), was synthesized as a structural model. Ag2O (0.02 g.-mole) and 0.01 g.-mole 2,2,6,6-tetramethyl-4-piperidone hydroxide refluxed 1 hr. in 25 ml. dry Et2O yielded 74.7% I, m. 36.degree., 5.9 .times. 1023 spins/mole. The high kinetic stability and the electron spin resonance (e.s.r.) spectrum of I were discussed. I (0.01 g.-mole) in 5 ml. MeOH added to 0.01 g.-mole HONH2 in 5 ml. MeOH yielded 51% I oxime, m. 180.degree., 6.0 .times. 1023 spins/mole. I (0.01 g.-mole) in H2O poured into satd. aq. H2NNHCONH2.HCl, and excess concd. aq. K2CO3 added gave the semicarbazone, m. 215.degree. (decompn.) (abs. alc.), with e.s.r. spectrum similar to that of I. I similarly gave a 2,4-dinitrophenylhydrazone, m. 250.degree. (decompn.), e.s.r. spectrum similar to that of I. I catalytically reduced with PtO2, Pd-C, or Raney Ni gave a substituted diamagnetic hydroxylamine, also obtained according

to Lehmann (Ber. 30, 2736(1897)) from phorone and H2NOH. The specific reactivity of the ketone group was particularly evident from the ready rearrangement of I with excess HONH2.HCl. Satd. aq. I (1.7 g.) treated at 20.degree. with 0.76 g. HONH2.HCl in a min. of H2O yielded 30% 2,2,7,7-tetramethyl-5-homopiperazinone (Ia) nitrogen oxide (II), m. 162.degree. (Et2O), 5.8 .times. 1023 spins/mole. Catalytic amts. of trylon-B and Na2WO4.2H2O in 5 ml. H2O and 3 ml. 30% H2O2 added to 3.4 g. Ia (m. 147-8.degree.) in 3 ml. warm MeOH and the mixt. kept 16 hrs. yielded 70% II. II is a rose-colored cryst. paramagnetic compd. with 2 possible conformational isomers. I (0.3 g.-mole) in 100 ml. dry Et2O added slowly to EtMgBr (24 g. Mg, 109 g. EtBr) in 220 ml. dry Et2O, the mixt. refluxed gently 4 hrs. (H2O-free conditions), and kept 16 hrs. gave 48 g. I and 2,2,6,6-tetramethyl-4-ethyl-4-piperidinol (IIa) nitrogen oxide (III). Na2WO4.2H2O (15 mg.), 20 mg. trylon-B, 4 ml. H2O, and 2 ml. 30% H2O2 added to 1.85 g. IIa (m. 66.degree.) in 4 ml. H2O, the mixt. kept 24 hrs. at 20.degree., satd. with solid K2CO3, extd. with Et2O, and the product submitted to thin-layer chromatography on Al2O3 in 1:1 MeOH-Me2CO gave the same chromatographic const. as that of III. The paramagnetic redn. product of the oxo group was obtained by decompn. of the hydrazone radical by the Kizhner method (Rozantsev, et al., CA 58, 10165e). I (0.06 g.-mole), 9.5 g. 85% N2H4.H2O, and 15 g. KOH in 100 g. (HOCH2CH2)2O refluxed 1.5 hrs. at 150.degree., the mixt. distd. into a liquid N-cooled receiver, and the distillate repeatedly extd. with Et2O yielded 8% 2,2,6,6-tetramethylpiperidine (IIIa) nitrogen oxide (IV), m. 38-9.degree.. Na2WO4.2H2O (15 mg.), 10 mg. trylon-B, and 1.5 ml. 30% H2O2 in 5 ml. H2O added to 70 g. IIIa in 5 ml. MeOH and the mixt. kept 2 days yielded 70.5% IV. The extremely high volatility and strongly camphor-like smell may be accounted for by the globular stereometry of IV.

L15 ANSWER 2376 OF 2379 CA
AN 60:26203 CA
OREF 60:4661g-h,4662a-b
TI Ganglioplegic activity of di-tert-butyl nitroxide, a stable free radical, and analog
AU Cummings, J. R.; Grace, J. L.; Latimer, C. N.
CS Am. Cyanamide Co., Pearl River, NY
SO J. Pharmacol. Exptl. Therap. (1963), 141(3), 349-55
DT Journal
LA Unavailable
AB Oral and intravenous hypotensive activities of di-tert-butyl nitroxide (tert-Bu2NR: R = O) (I) and 15 analogs were studied in rats, dogs, and cats. Nine of the compds. tested lowered the blood pressure and also significantly augmented the vasopressor responses to intravenous epinephrine and phenylethylamine. An immediate hypotensive response was evoked by the free radicals I and 2,2,6,6-tetramethylpiperidine N-oxide and by the corresponding hydroxy hydrochlorides, showing that the presence of the free electron did not confer unusual pharmacol. activity. Bladder contractions to stimulation of the preganglionic pelvic nerve fibers were blocked by I intraarterially and, in smaller doses, intravenously. I also blocked contractions elicited by intraarterial dimethylphenylpiperazinium but the increased tone produced by intraarterial serotonin was either unaffected or augmented. A similar effect was produced by II (R = OH). The increases in heart rate and contractile force produced by preganglionic stimulation of stellate cardiac nerve were blocked by I but the same effects resulting from postganglionic stimulation were unaffected. Enhanced salivary flow induced by pilocarpine was unaltered by I. The normal postganglionic potentials produced by stimulation of preganglionic fibers of the superior cervical sympathetic nerve in cats were blocked by both I and II, as were the contractions of the nictitating membrane. The effects of postganglionic stimulation were unaffected. Acute toxicities of I and II (L.D..50, mg./kg., oral and intravenous) were I, 505, 53.8 and II, 222, 49.7. Dogs given 20 times the ganglioplegic dose orally showed symptoms due to ganglionic blockade (mydriasis, loss of accomodation, and nictitating membrane relaxation) but no deaths.

L15 ANSWER 2377 OF 2379 CA COPYRIGHT 2003 ACS
AN 59:66851 CA
OREF 59:12330b-c
TI Nature of the spin-lattice interaction in magnetically diluted free radicals
AU Il'yasov, A. V.; Garif'yanov, N. S.; Timerov, R. Kh.
CS Phys.-Tech. Inst., Kazan Branch Acad. Sci. U.S.S.R., Kazan
SO Dokl. Akad. Nauk SSSR (1963), 150(3), 588-91
DT Journal
LA Unavailable
AB The electron paramagnetic resonance was studied for solid (supercooled) solns. of the free radicals of .alpha.,.alpha.-diphenyl-.beta.-picrylhydrazyl and of 2,2,6,6-tetramethylpiperidine oxide in EtOH, MeOH, C6H6, PhMe, and in mixts. of the alc. with glycerol and with H2O. The results show that the time for spin-lattice relaxation in the supercooled solns. with a radical concn. of 10-210-3M is independent of concn. and the nature of the solvent. The mechanism for the spin-lattice interaction is greatly dependent on the type of solvent.

L15 ANSWER 2378 OF 2379 CA COPYRIGHT 2003 ACS
AN 59:6672 CA
OREF 59:1204f-g
TI Electron spin resonance (E.S.R.) in liquid and supercooled solutions of some free radicals
AU Garif'yanov, N. S.; Il'yasov, A. V.; Yablokov, Yu. V.
CS Phys. Tech. Inst. Branch Acad. Sci., U.S.S.R., Kazan
SO Dokl. Akad. Nauk SSSR (1963), 149, 876-9
DT Journal
LA Unavailable
AB The effect of the immediate environment of the mol. spin on the parameters of the hyperfine structure of .alpha.,.alpha.-diphenyl-.beta.-picrylhydrazyl and of 2,2,6,6-tetramethylpentamethylene N oxide was studied. The measurements were made at 9320 Mc. in the temp. range 77-300.degree.K. MeOH, EtOH, glycerol-MeOH mixt., C6H6, toluene, CHCl3, and polystyrene dild. with C6H6 or CHCl3 were used as solvents. Equations are derived for describing the E.S.R. spectrum in liquid and supercooled solns. A table of the parameters of the hyperfine structure shows that the ratio of the consts. of the isotropic hyperfine structure depends noticeably on the solvent. Measurement of the g factor of tetramethylpentamethylene N oxide at 36,000 Mc. showed that the anisotropy was not more than 0.002.

L15 ANSWER 2379 OF 2379 CA COPYRIGHT 2003 ACS
AN 58:44164 CA
OREF 58:7523d-e
TI Effect of solvents on the electron paramagnetic resonance (E.P.R.) spectra of some free radicals
AU Il'yasov, A. V.
SO Zh. Strukt. Khim. (1962), 3, 95-7
DT Journal
LA Unavailable
AB The effect of solvents on the E.P.R. spectra of the free radicals I and II was studied at a frequency of 9320 Mc. The width of the line, .delta.H, was measured as the distance between the max. and min. of the first deriv. of the absorption curve. I had .delta.H and sepn. of the components as follows: in C6H6, PhMe, and CHCl3, 7 and 15.6 oe.; in EtOH, 6.4 and 16.8 oe.; in 40% aq. EtOH, 4.4 and 18.9 oe. Similarly for II .delta.H and the sepn. were, resp.; in C6H6, PhMe, and CHCl3, 6 and 11.7 oe.; and in EtOH + 30% H2O, 4 and 13 oe. The difference in .delta.H for the aq. solns. is not due to viscosity, because heating to 40.degree. to equalize the viscosities does not remove the difference. The phenomenon may be caused by the presence of a solvation shell around the free radicals.

=> d l11 139-144 bib,ab

L11 ANSWER 139 OF 144 CA COPYRIGHT 2003 ACS
AN 92:102211 CA
TI Photolysis of alkylcobaloximes, methylsalen, cobalamines and coenzyme B12 in protic solvents: an ESR and spin-trapping technique study
AU Maillard, Ph.; Giannotti, C.
CS Inst. Chim. Subst. Nat., CNRS, Gif sur Yvette, 91190, Fr.
SO Journal of Organometallic Chemistry (1979), 182(2), 225-37
CODEN: JORCAI; ISSN: 0022-328X
DT Journal
LA English
AB The photolysis in protic solvents in the visible region >420 nm of several alkylcobaloximes, cobalamines, methylsalen, and coenzyme B12 was studied by ESR spectroscopy including the use of spin-trapping techniques involving 5,5'-dimethylpyrroline-N-oxide (DMPO), phenyl-N-tert-butylnitrone, and .alpha.-4-pyridyl-1-oxide-N-tert-butylnitrone. During the photolysis, H atoms are probably abstracted from the C(10) position of the corrin equatorial ligand in the case of coenzyme B12 and cobalamine derivs. The ESR spectra of the anaerobic photolysis of alkylcobaloximes in aprotic solvents in the presence of DMPO, followed by the addn. of air or pure O, provide evidence for intraligand radical spin-trapping reactions.

L11 ANSWER 140 OF 144 CA COPYRIGHT 2003 ACS
AN 92:31909 CA
TI Spin trapping and electron spin resonance detection of radical intermediates in the photodecomposition of water at titanium dioxide particulate systems
AU Jaeger, Calvin D.; Bard, Allen J.
CS Dep. Chem., Univ. Texas, Austin, TX, 78712, USA
SO Journal of Physical Chemistry (1979), 83(24), 3146-52
CODEN: JPCHAX; ISSN: 0022-3654
DT Journal
LA English
AB Spin trapping and ESR detection [with the traps .alpha.-Ph N-tert-Bu nitrone and .alpha.-(4-pyridyl N-oxide) N-tert-Bu nitrone] were used to detect free-radical intermediates formed during in situ irradn. of TiO2 and platinized TiO2 powders in aq. solns. Evidence for the prodn. of OH radical (formed in the oxidn. of H2O) and HO2 radical (probably formed in a redn. step) is presented. Other spin adducts, attributed to decompn. reactions of the spin traps, were also found. The results suggest an important role for photogenerated OH in many photocatalytic and photosynthetic processes using TiO2 powders.

L11 ANSWER 141 OF 144 CA COPYRIGHT 2003 ACS
AN 92:2794 CA
TI Spin trapping of superoxide
AU Finkelstein, Eli; Rosen, Gerald M.; Rauckman, Elmer J.; Paxton, John
CS Med. Cent., Duke Univ., Durham, NC, 27710, USA
SO Molecular Pharmacology (1979), 16(2), 676-85
CODEN: MOPMA3; ISSN: 0026-895X
DT Journal
LA English
AB Due to conflicting reports in the literature, the spin trapping of superoxide by the nitrone 5,5-dimethyl-1-pyrroline N-oxide (I) was reinvestigated. Superoxide could indeed be trapped and the 5,5-dimethyl-2-hydroxylpyrrolidinoxyl (II) and 5,5-dimethyl-2-hydroperoxypyrrolidinoxyl (III) adducts were distinguishable. In contrast to earlier work, III was highly unstable and decompd. into II. 2,5,5-Trimethyl-1-pyrroline N-oxide (TMPO), a spin trap structurally similar to I but lacking a .beta.-hydrogen, formed a stable nitroxide upon reaction with superoxide. Rate consts. for the reaction of superoxide

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     with I and TMPO were measured and indicated that spin trapping was an
     extremely inefficient method for the detection of superoxide.  The new
     spin trap, .alpha.-4-pyridyl-1-oxide-N-tert-butyl nitrone (POBN), was also
     capable of distinguishing the difference between superoxide and hydroxyl
     radical.  The nitroxide formed upon the reaction of superoxide with POBN
     was unstable.

L11  ANSWER 142 OF 144  CA  COPYRIGHT 2003 ACS
AN   91:108398  CA
TI   The ESR spin trapping chemistry of .alpha.-(4-pyridyl 1-oxide)
     N-tert-butyl nitrone and poly(phenyl-N-tert-butyl nitrone)
AU   Wang, Yeeyang
CS   Univ. Georgia, Athens, GA, USA
SO   (1978) 194 pp.  Avail.: Univ. Microfilms Int., Order No. 7914065
     From: Diss. Abstr. Int. B 1979, 39(12, Pt. 1), 5961
DT   Dissertation
LA   English
AB   Unavailable

L11  ANSWER 143 OF 144  CA  COPYRIGHT 2003 ACS
AN   90:5442  CA
TI   On spin trapping hydroxyl and hydroperoxy radicals
AU   Janzen, Edward G.; Nutter, Dale E., Jr.; Davis, Edward R.; Blackburn,
     Barry J.; Poyer, J. Lee; McCay, Paul B.
CS   Guelph-Waterloo Cent. Grad. Work Chem., Univ. Guelph, Guelph, ON, Can.
SO   Canadian Journal of Chemistry (1978), 56(17), 2237-42
     CODEN: CJCHAG; ISSN: 0008-4042
DT   Journal
LA   English
AB   The assignments of the HO.bul., HOO.bul., and ROO.bul. (R = alkyl) radical
     spin adducts of PhCH:N+(O-)CMe3 are discussed.  Results of new expts.
     designed to test the assignments of the first two are described.  The
     HO.bul. adduct assignment is correct.  The relation between a.beta.Hand aN
     is detd. and gives a good fit for the 7 pairs of consts. reported for the
     HO.bul. adduct.  The assignments for the HOO.bul. adduct may be correct
     but no proof is available.

L11  ANSWER 144 OF 144  CA  COPYRIGHT 2003 ACS
AN   89:23427  CA
TI   Spin trapping with .alpha.-pyridyl 1-oxide N-tert-butyl nitrones in
     aqueous solutions.  A unique electron spin resonance spectrum for the
     hydroxyl radical adduct
AU   Janzen, Edward G.; Wang, Y. Y.; Shetty, Raghav V.
CS   Guelph Waterloo Cent. Grad. Work Chem., Univ. Guelph, Guelph, ON, Can.
SO   Journal of the American Chemical Society (1978), 100(9), 2923-5
     CODEN: JACSAT; ISSN: 0002-7863
DT   Journal
LA   English
AB   The spin-trapping chem. of .alpha.-4-pyridyl 1-oxide N-tert-Bu nitrones is
     described in aq. soln. as applied to the problem of detecting HO.bul.
     radical.  Hyperfine-splitting consts. of the hydroxyl adduct are shown as
     a function of pH and H2O2 concn.  A 2nd set of doublets was obsd. which
     were attributed to a 2nd nitroxide; this may be the hydroperoxy adduct,
     but this assignment is unsupported at this time.


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L13        1014 L3

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     FILE 'REGISTRY' ENTERED AT 11:12:06 ON 03 MAY 2003
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